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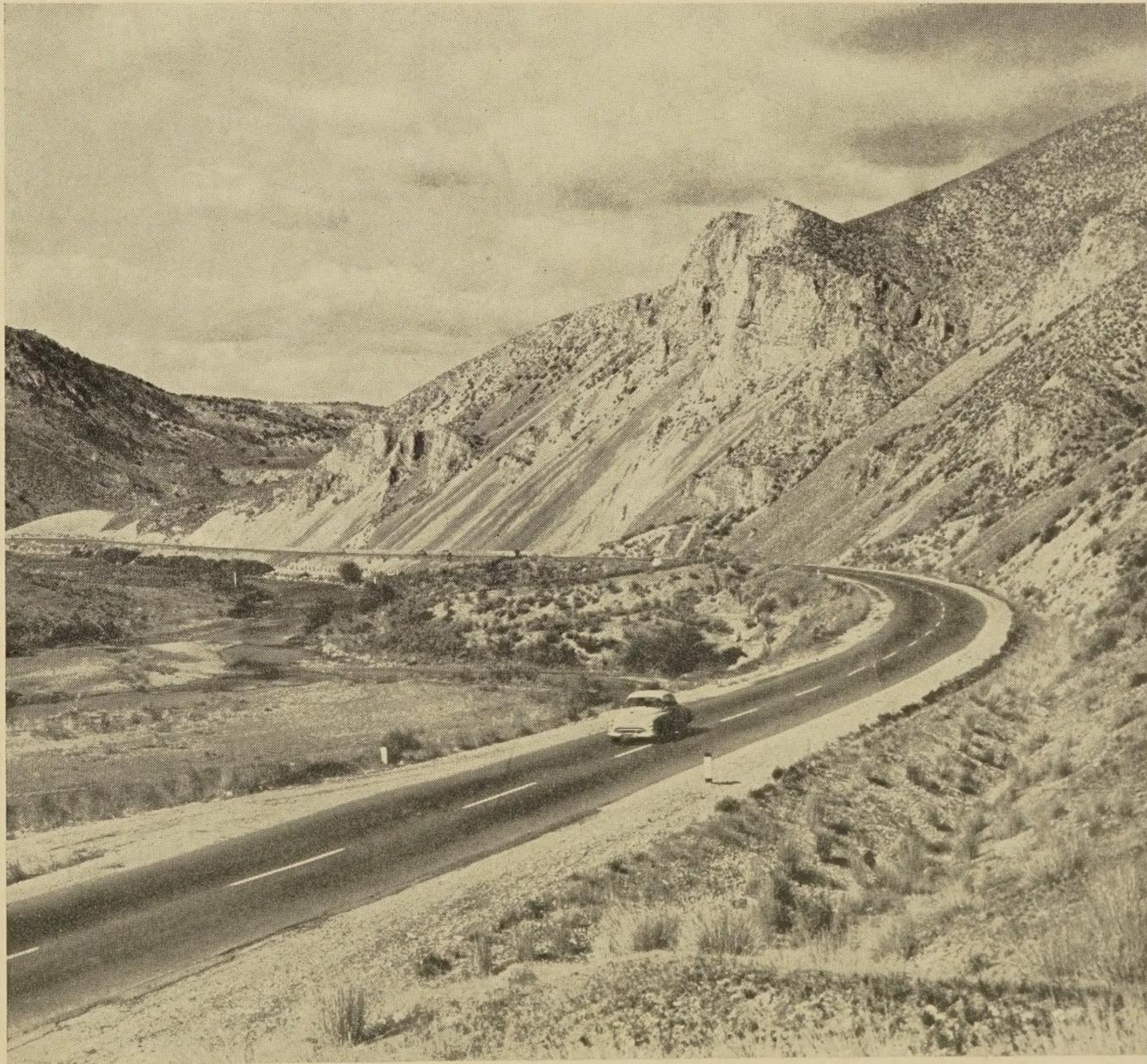
AUGUST 1954

# Public Roads

A JOURNAL OF HIGHWAY RESEARCH



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# Heat-Resistant Properties of Additives for Bituminous Materials

BY THE PHYSICAL RESEARCH BRANCH  
BUREAU OF PUBLIC ROADS

Reported by PAUL F. CRITZ, Supervising  
Highway Physical Research Engineer

*In past years, it was generally believed that hot-mixed, hot-laid bituminous pavements were not subject to film stripping in the presence of moisture. Investigations made where failures of such bituminous pavements have occurred indicated, however, that film stripping had taken place in the underlying courses of the pavements, the condition being more severe in the case of impermeable bases than in those having porous granular materials. To counteract this deficiency, the use of commercial additives for treating asphalt was adopted. The results were not always satisfactory. It was felt by many authorities that the additives might not be stable at the temperatures used in constructing hot-mix bituminous pavements or when held at relatively high temperatures during shipment from the refinery. As a result, manufacturers undertook the production of additives having heat-resistant qualities for the treatment of asphalt.*

*This study was undertaken to test the heat-resistant properties of three additives. It was found that prolonged heating of treated asphalt at high temperatures prior to mixing reduced the effectiveness of the additives as measured by the immersion-compression test method, and that it was more detrimental to small masses than to large ones. Overheating the aggregate prior to mixing reduced the effectiveness even more. With few exceptions, the additives were found to be effective in increasing the percentage of retained strength and in reducing moisture absorption and volumetric swell. If proper attention is given to the control of temperatures of asphalt and especially the aggregate, the additives studied may be expected to reduce the detrimental effect of moisture absorption.*

THE value of certain chemical agents as aids in coating aggregates with bituminous materials and increasing the resistance of the bituminous film to stripping has been fairly well established and accepted for cold-mix and cold-laid types of mixtures. Laboratory studies<sup>1</sup> and field investigations have shown that a number of such agents are of considerable benefit for the treatment of liquid asphaltic materials to be used with hydrophilic aggregate or with wet aggregate.

The possible value of these additives or the necessity for using them in hot-mixed bituminous mixtures was not considered in the earlier studies because, at that time, the general feeling existed that mixtures prepared hot with viscous bituminous materials, such as asphalt cement, were not liable to film stripping in the presence of moisture. However, investigations of instances of unsatisfactory service behavior of bituminous pavements revealed that extensive film stripping has occurred in many cases in the underlying courses of hot-mix, hot-laid bituminous pavements. This condition was found most often in courses laid on impermeable bases such as portland cement concrete or brick

pavement, and less frequently in pavements laid on porous granular bases. To counteract this unsatisfactory performance, additives have also been used for treating asphalt for use in the hot-mix types of asphaltic pavements. Generally, the additives are added to the asphalt at the refinery.

So far, their use in hot-plant mixtures has not always been satisfactory. The results have been variable in many cases and often inconsistent. On one important experimental project, several additives that had shown value in laboratory studies were used. Later examination of the pavement indicated that stripping had occurred and that it was more dependent on the type of aggregate and the type of base on which the surfacing was laid than on the additive used. Informal reports indicate that similar results have been obtained in other localities. The consensus of opinion has been that additives, as currently produced, may not be stable at the temperatures used in constructing hot-mix bituminous pavements or possibly they do not remain stable when held at relatively high temperatures during shipment from the refinery and prior to use on the job.

As a result of these developments, manufacturers undertook the production of additives for the treatment of asphalt that would be resistant to the heat to which they would

be subjected during shipment and plant mixing. Materials, for which such properties were claimed, were later placed on the market.

To determine to what extent these materials are heat resistant, the Bureau of Public Roads conducted a study in which asphalt cement treated with an additive, and asphalt cement not treated, were heated to two different temperatures and then combined with aggregates which also had been heated to two different temperatures. The resultant mixtures were then formed into test specimens which were subjected to the immersion-compression test.

## Conclusions

The results of this investigation, with respect to the three additives which were tested with asphalt from one source, are summarized as follows:

1. Prolonged heating of the treated asphalt at high temperatures prior to mixing reduced the effectiveness of the additives. For these tests, heating at 325° F. for 72 hours practically destroyed the effectiveness of the additives as measured by the results of the immersion-compression test. However, it is known that the effect of heating is much more detrimental to small masses of asphalt than to large ones. Therefore, it may be concluded that, for the same temperatures and periods of heating that were used in these tests, much less severe reductions in the effectiveness of the additives would take place in practice where the asphalt is held at elevated temperatures in large masses.

2. Overheating the aggregate reduced the effectiveness of the additives more than did prolonged heating of the treated asphalt prior to mixing. It may be noted that mixing operations, as performed in connection with this investigation, much more nearly simulated the conditions of practice than did the preliminary heating of the asphalt.

3. Even under the severe conditions of these tests the additives, with only a few exceptions, were effective in specimens subjected to the immersion-compression test in increasing the percentage of retained strength and in reducing the moisture absorption and volumetric swell.

It is concluded that if proper attention is given to the control of the temperature of the asphalt and the aggregate, particularly the

<sup>1</sup> *Additives for bituminous materials*, by P. F. Critz, PUBLIC ROADS, vol. 24, No. 5, July-Aug.-Sept. 1945; and *Further developments and application of the immersion-compression test*, by J. T. Pauls and J. F. Goode, PUBLIC ROADS, vol. 25, No. 6, Dec. 1948.

**Table 1.—Physical properties of asphalt cement with and without additives, before and after heating**

Additive in asphalt	Heating period		Physical property			Changes in physical properties	
	Temperature	Time	Penetration at 77° F.	Ductility at 77° F.	Softening point	Penetration	Softening point
None.....	°F. (1)	Hours	88	250+	118.0	-----	-----
A.....	(1)	-----	116	200+	112.5	-----	-----
B.....	(1)	-----	108	200+	113.4	-----	-----
C.....	(1)	-----	115	180	113.2	-----	-----
None.....	250	24	78	200+	121.0	-10	+3.0
A.....	250	24	98	200+	116.0	-18	+3.5
B.....	250	24	91	200+	117.6	-17	+4.2
C.....	250	24	100	200+	116.5	-15	+3.3
None.....	250	48	71	200+	124.3	-17	+6.3
A.....	250	48	89	200+	117.0	-27	+4.5
B.....	250	48	80	200+	119.8	-28	+6.4
C.....	250	48	86	200+	119.5	-29	+6.3
None.....	250	72	63	200+	125.5	-25	+7.5
A.....	250	72	83	200+	119.0	-33	+6.5
B.....	250	72	69	200+	125.0	-39	+11.6
C.....	250	72	77	190	123.0	-38	+9.8
None.....	325	24	70	200+	125.0	-18	+7.0
A.....	325	24	78	200+	121.0	-38	+8.5
B.....	325	24	74	185	121.8	-34	+8.4
C.....	325	24	80	200+	120.8	-35	+7.6
None.....	325	48	54	200+	130.3	-34	+12.3
A.....	325	48	61	200+	129.0	-55	+16.5
B.....	325	48	56	200+	129.0	-52	+15.6
C.....	325	48	63	200+	128.3	-52	+15.1
None.....	325	72	32	17	149.8	-56	+31.8
A.....	325	72	59	200+	130.0	-57	+17.5
B.....	325	72	33	17	150.0	-75	+36.6
C.....	325	72	33	17	150.3	-82	+37.1

1 Original test—before subjecting to heat.

latter, the additives studied in this investigation may be expected to reduce the detrimental effects of moisture in hot-mixed asphaltic pavements containing hydrophilic aggregates.

**Plan of the Experiment**

Three commercial additives, designated in this report as A, B, and C, respectively, were used with an asphalt cement of 88 penetration

in asphaltic concrete mixtures which were subjected to the immersion-compression test (ASTM test method D1075-49T). For comparative purposes, mixtures identical with these, except that they contained no additives, were also tested.

The immersion-compression test measured the loss of cohesion resulting from the action of water on compacted bituminous mixtures. An index of the loss was obtained by com-

paring the compressive strength of specimen that were immersed in water with that of specimens which were not immersed.

The asphalt cement, with and without additives, was subjected to two different temperatures, 250° and 325° F., and was held at those temperatures for periods of 0, 24, 48 and 72 hours prior to the mixing operation. The aggregate was crushed granite having definitely hydrophilic properties. The amount of additive used in all instances was 2 percent by weight of the bituminous material. The basic properties of the asphalt cement with and without additives as well as the change in these properties resulting from heating are given in table 1. The composition of the mixture used throughout this study is given below. It was designed to produce compressed specimens containing an appreciable percentage of voids in order to insure a high degree of moisture absorption since the chief property claimed for additives is that they increase the resistance of the coated aggregate to the detrimental effects of moisture.

**Materials:**

Aggregate: 100 percent crushed granite

Bitumen: Asphalt cement of 88 penetration.

Additives: Designated as A, B, and C.

**Table 2.—Temperatures at which molded specimens were heated**

Series	Temperature, ° F.	
	Asphalt	Aggregate
I.....	250	300
II.....	250	400
III.....	325	300
IV.....	325	400

**Table 3.—Results of tests of specimens with and without additives**

Heating period	Dry strength				Wet strength				Retained strength				Moisture absorption				Volumetric swell			
	No additive	A	B	C	No additive	A	B	C	No additive	A	B	C	No additive	A	B	C	No additive	A	B	C
SERIES I: ASPHALT TEMPERATURE 250° F., AGGREGATE TEMPERATURE 300° F.																				
Hours	P.s.i.	P.s.i.	P.s.i.	P.s.i.	P.s.i.	P.s.i.	P.s.i.	P.s.i.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.
0.....	286	290	300	298	107	200	205	201	37.4	69.0	68.3	67.4	4.2	2.7	3.1	3.0	2.1	0.7	1.2	1.3
24.....	284	341	293	291	124	195	209	182	43.7	57.2	71.3	62.5	3.6	2.6	3.0	2.8	1.8	0.6	0.9	1.2
48.....	287	320	312	317	95	224	224	159	33.1	70.0	71.8	50.2	3.9	2.7	3.1	3.0	3.2	0.9	1.2	1.8
72.....	340	344	353	316	135	214	237	167	39.7	62.2	67.2	52.8	3.4	2.6	2.9	3.2	1.9	0.7	1.4	1.7
SERIES II: ASPHALT TEMPERATURE 250° F., AGGREGATE TEMPERATURE 400° F.																				
0.....	331	345	347	354	176	263	257	258	53.2	76.2	74.1	72.9	3.3	2.4	2.8	2.6	1.0	0.5	0.9	1.1
24.....	312	350	323	321	165	235	249	225	52.9	67.2	75.9	70.1	2.9	2.7	2.8	2.3	1.1	0.9	0.6	0.8
48.....	332	346	354	327	207	261	240	248	62.4	75.4	67.8	75.8	2.5	2.5	2.9	2.3	1.4	0.8	0.9	1.5
72.....	390	360	415	359	243	244	323	230	62.3	67.8	77.8	64.1	2.6	2.2	2.6	2.8	0.8	0.4	0.9	1.2
SERIES III: ASPHALT TEMPERATURE 325° F., AGGREGATE TEMPERATURE 300° F.																				
0.....	280	308	322	320	113	216	207	198	40.4	70.2	64.3	61.9	4.1	2.6	3.0	3.0	2.0	0.8	1.1	1.4
24.....	314	356	330	322	140	220	230	210	44.6	61.8	69.7	65.2	3.3	2.5	2.7	2.1	1.7	0.6	0.8	0.6
48.....	386	382	384	357	162	265	229	230	42.0	69.4	59.6	64.4	3.0	2.5	3.1	2.2	2.6	1.0	1.3	1.7
72.....	505	404	565	478	290	250	291	280	57.4	61.9	51.5	58.6	2.7	2.3	3.0	3.0	1.4	0.5	1.7	1.6
SERIES IV: ASPHALT TEMPERATURE 325° F., AGGREGATE TEMPERATURE 400° F.																				
0.....	308	358	358	377	179	267	262	300	58.1	74.6	73.2	79.6	3.2	2.2	2.5	2.2	1.2	0.5	0.8	0.7
24.....	329	385	357	366	215	266	301	268	65.3	69.1	84.4	73.2	2.6	2.4	2.3	1.9	0.8	0.6	0.3	0.4
48.....	425	424	402	414	280	334	305	288	65.9	78.8	75.9	69.6	1.8	2.1	2.5	2.0	1.0	0.6	1.0	1.0
72.....	600	440	640	548	390	311	463	385	65.0	70.7	72.4	70.2	2.3	2.1	2.5	2.2	0.6	0.3	0.8	1.2

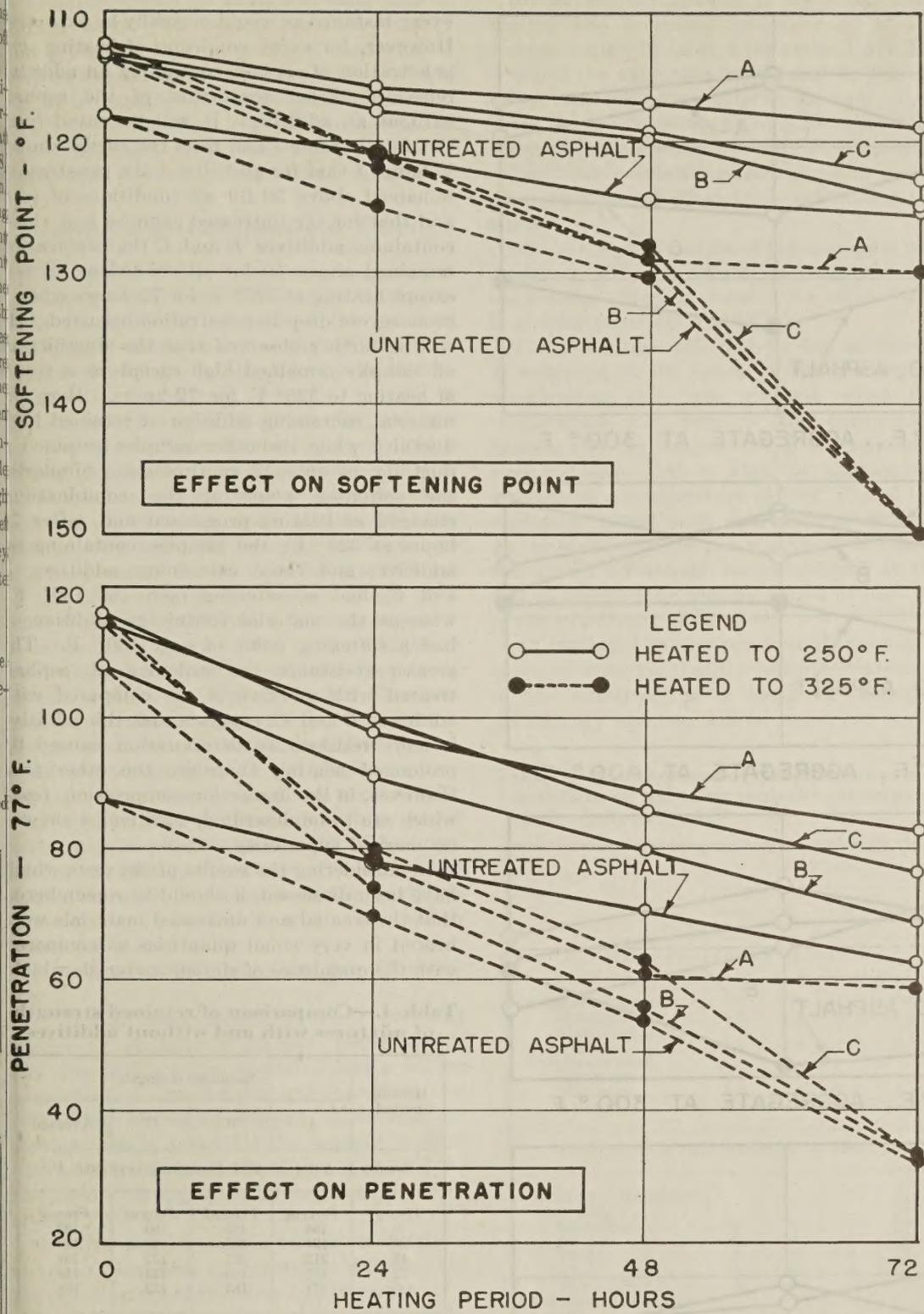


Figure 1.—Effect of heat upon properties of asphalt cement with and without additives.

Proportions:  
 Aggregate; gradation:

Sieve size	Percent passing
3/8-inch	100
No. 4	82
No. 10	36
No. 40	13
No. 80	9
No. 200	4

Bitumen: 6 percent, by weight of aggregate.  
 Additives: 2 percent by weight of asphalt.  
 Physical properties of preliminary test specimens, without additives, prepared according to standard procedure:

Density: Theoretical maximum, 2.518; actual, 2.274.  
 Voids: In aggregate, 21.2 percent; in compressed mixture, 9.7 percent.  
 Moisture absorption, 3.9 percent.  
 Volumetric swell, 2.2 percent.  
 Compressive strength, dry, 278 p.s.i.  
 Retained strength of immersed specimens, 43 percent.

It was obviously not possible in the laboratory to duplicate normal conditions where a large mass of material is held at a high temperature in a tank car or storage tank with a relatively small surface exposure. In this investigation the asphalt was heated in tinned containers 3 inches in diameter and 2

inches deep. Each tin contained 105 grams of asphalt, slightly more than the amount required for a single batch for two specimens. The containers were covered with loose-fitting lids to reduce oxidation. At the end of the heating period the bituminous material was immediately mixed with the aggregate which also had been brought to the desired temperature.

Two temperatures were selected to which the aggregates were heated immediately prior to mixing. They were 300° and 400° F. The higher temperature is somewhat above that recommended for good construction procedure but it is not as high as the temperature to which aggregates are often heated unless strict control is exercised. All aggregate was held overnight at 300° F. which was the heating limit of the oven. Aggregate to be used at 400° F. was raised from 300° to 400° F. on a hot plate with continuous stirring to obtain uniformity and eliminate local overheating.

Mixing was completed mechanically as rapidly as possible and from each batch, two 3-inch by 3-inch test cylinders were molded in accordance with the standard procedure used in the immersion-compression test. Three batches or six test specimens constituted one test unit. Three specimens were tested dry and three were tested after immersion in water at 120° F. for four days. The two groups of three specimens each were so selected that the average density of the specimens selected for immersion was, as nearly as possible, the same as that of the specimens to be tested dry.

The results reported are the compressive strengths of the specimens tested dry, the compressive strengths of the specimens tested wet after immersion, and the strength retained by the wet specimens expressed as a percentage of the strength of the comparable dry specimens.

For reference purposes, the specimens subjected to the four different heating temperatures are grouped into four series as shown in table 2.

No attempt was made to determine the composition of the additives. A variety of materials are available which can be used in their preparation and substitutions can be made whenever the producers find it advisable for economic or other reasons. Therefore, the actual composition of the additives used in this study is of little concern and any conclusions based upon their composition would be of no value if additives produced more recently had a different composition. Data presented and conclusions drawn herein consequently are based upon the tests of the additives made available for the study.

**Effect of Additives on Asphalt**

The asphalt cement used in this study had an original penetration of 88, a ductility of 250 (plus) and a softening point of 118° F. As shown by the data in table 1 and the curves in figure 1, these properties were changed when additives were combined with asphalt. For example, with 2 percent of additive A, the

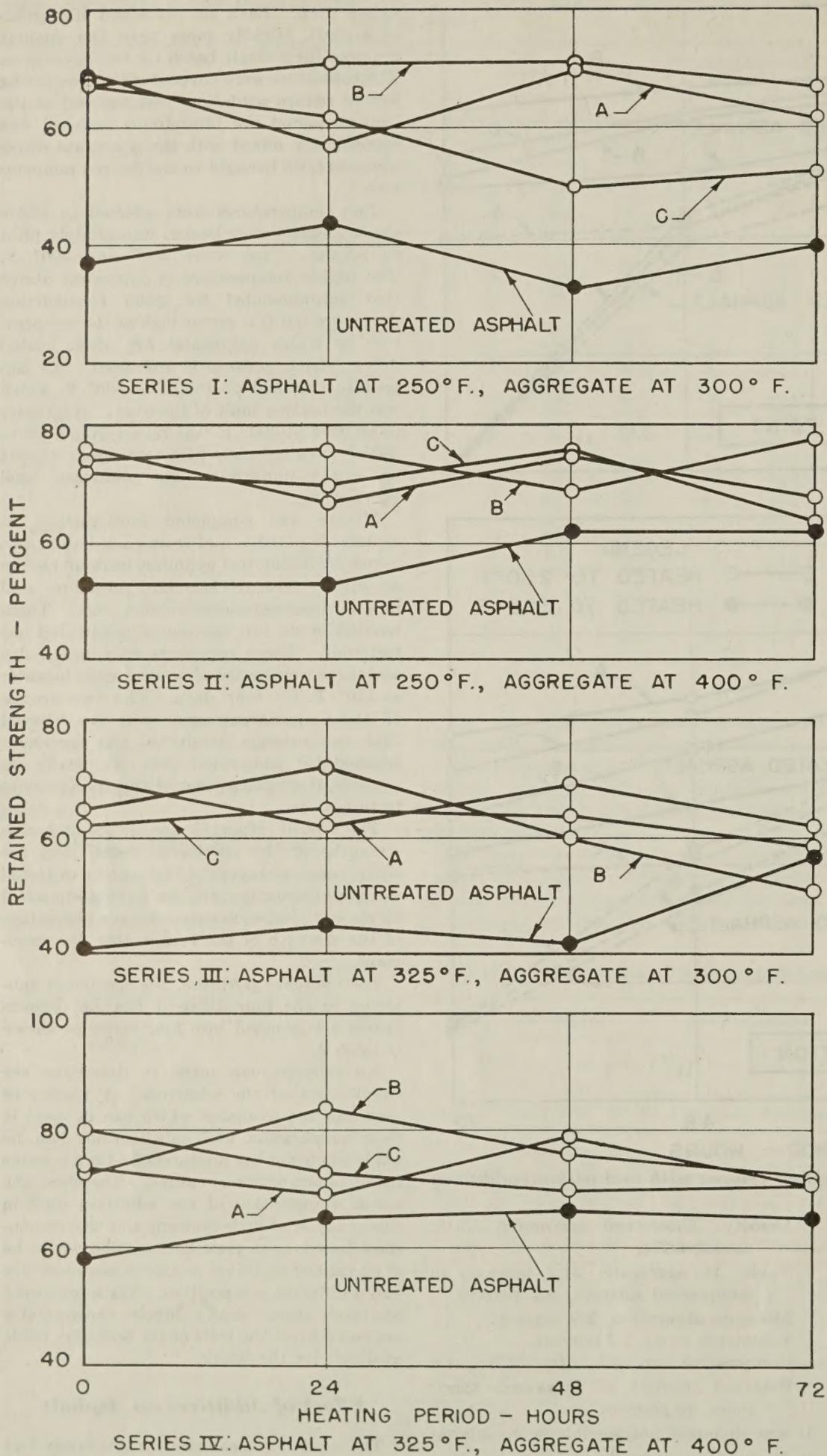


Figure 2.—Compressive strength retained after four days immersion at 120° F.

penetration was increased to 116; with additive B, it was increased to 108; and with additive C, it was increased to 115.

As the period of heating was extended and also as the heating temperature was raised, the penetration of the asphalt dropped in

every instance as would normally be expected. However, for every condition of heating, the penetration of asphalt containing an additive remained higher than that of the asphalt without an additive. It will be noted from the data in table 1 and from the curves shown in figure 1 that for additive A the penetration remained above 50 for all conditions of test, and that for the untreated samples and those containing additives B and C the penetration remained above 50 for all conditions of test except heating at 325° F. for 72 hours when a more severe drop in penetration occurred. It will be further observed that the ductility of all samples remained high except as a result of heating to 325° F. for 72 hours. Here the material containing additive A retained high ductility while the other samples retained a ductility of only 17 centimeters. Similarly, the softening point of the combinations changed as heating progressed and, after 72 hours at 325° F., the samples containing no additive, and those containing additives B and C, had a softening point of 150° F., whereas the material containing additive A had a softening point of only 130° F. The greater resistance to hardening of asphalt treated with additive A, as compared with additives B and C, suggests that this additive is more resistant to deterioration caused by prolonged heating than are the other two. However, in the immersion-compression tests, which are to be described, additive A showed no marked superiority.

In considering the results of the tests which have been discussed, it should be remembered that the treated and untreated materials were heated in very small quantities as compared with the quantities of similar materials which,

Table 4.—Comparison of retained strengths of mixtures with and without additives

Heating period	Retained strength <sup>1</sup>			
	A	B	C	Average
SERIES I: ASPHALT 250° F. AGGREGATE 300° F.				
Hours	Percent	Percent	Percent	Percent
0	184	183	180	182
24	131	163	143	146
48	212	217	152	194
72	157	169	133	153
Avg.	171	183	152	168
SERIES II: ASPHALT 250° F. AGGREGATE 400° F.				
0	143	139	137	140
24	127	143	132	134
48	121	109	122	117
72	109	125	103	112
Avg.	125	129	124	126
SERIES III: ASPHALT 325° F. AGGREGATE 300° F.				
0	174	159	153	162
24	139	156	146	147
48	165	142	153	153
72	108	90	102	100
Avg.	147	137	139	141
SERIES IV: ASPHALT 325° F. AGGREGATE 400° F.				
0	128	126	137	130
24	106	129	112	116
48	120	115	106	114
72	109	111	108	109
Avg.	116	120	116	117

<sup>1</sup> Expressed as percentage of retained strength of mixture without additives.

n practice, would be heated in tank cars or storage tanks. The results show very clearly that prolonged heating produces hardening of both untreated and treated asphalts, but it is not to be expected that the hardening which would occur in tank cars or storage tanks would approach in severity the hardening which took place in the small test samples.

### Immersion-Compression Tests

The results of the immersion-compression tests are given in table 3 and are shown graphically in figure 2. In table 4 the retained strengths of mixtures containing additives are compared with the retained strengths of mixtures without additives.

Reference to table 3 shows that, as the severity of the test increased, there was a general increase in the compressive strength of the specimens, both those which were tested dry and those which were tested wet after having been immersed in water for four days at a temperature of 120° F. This is attributed to the hardening of the asphalt which took place during the preliminary heating, as shown in table 1 and figure 1, and to the additional hardening which took place during the mixing operation when the asphalt in thin films was brought in contact with the hot aggregates. As has been stated, the hardening of the asphalt which took place in the laboratory during the preliminary heating was undoubtedly much greater than the hardening which would take place in practice as a result of the preliminary heating. However, the additional hardening which took place during mixing is more nearly comparable with that which would occur in practice.

A study of tables 3 and 4 and figure 2 shows that:

1. For all test conditions except one, the specimens containing asphalt treated with any one of the three additives had a higher percentage of retained strength than did those containing untreated asphalt. The one exception was in series III for asphalt treated with additive B, heated for 72 hours at 325° F., and used with aggregate having a temperature of 300° F.

2. With respect to percentage of retained strength, there was no consistent difference in the performance of the three additives.

3. In general, the effectiveness of the additives, as measured by percentage of retained strength, was reduced:

(a) As the period of preliminary heating was increased.

(b) As the temperature of the preliminary heating was increased. Comparison of the average values in table 4 for series I and III in which the aggregate was heated to 300° F. shows that this effect is quite marked. It is much less so in series II and IV, where the aggregate temperature was 400° F., thus indicating that overheating the aggregate had a more detrimental effect than overheating the asphalt.

(c) As the temperature of the aggregate was increased. This is shown by comparison of the average values in table 4 for series I and II and for series III and IV.

The advantage afforded by the additives, as measured by the results of the immersion-compression test, was greatest when the temperature and time of the preliminary heating and the temperature of the aggregate were the least; that is, when the asphalt was brought to a temperature of 250° F. and immediately mixed with aggregate at 300° F. As the severity of the test conditions increased this initial advantage decreased until at the end of the 72-hour heating period at 325° F., it was practically eliminated.

As indicated in the previous discussion, it is fair to conclude that the loss in effectiveness of the additives, as a result of prolonged preliminary heating, will be much less severe

in practice than it was in these laboratory tests. It is also concluded that overheating the aggregate is more detrimental than prolonged heating of the asphalt.

### Effect of Additives on Bituminous Mixtures

All specimens used in this study had the same composition and were prepared and tested under a single procedure. An opportunity is afforded, therefore, to consider the effects of additives upon certain properties of the mixture, such as moisture absorption and volumetric swell.

#### Moisture absorption

Reference to the data given in table 3 shows that with few exceptions moisture absorption was less when additives were used than when the asphalt was untreated. The data show that only general relations can be observed with respect to the effect of additives on absorption. A further comparison of the relative effectiveness of the additives is shown in table 5. Of the three additives tested, all were of some benefit in reducing moisture absorption and additive A was the most effective in this respect.

#### Volumetric swell

Experience has indicated that volumetric swell of bituminous mixtures subjected to moisture may be expected to result in loss of stability or strength. Consequently, it would be expected that additives would reduce the tendency of mixtures to swell if they were to be effective in maintaining the structural stability of the mixtures. The changes in volume which occurred in the four-day immersion period are given in table 3, and are expressed as the percentage increase in volume on the basis of the volume of the specimens before immersion. It will be observed that, in general, the maximum swell recorded occurred in specimens that did not contain an additive and that for the same test conditions, the amount of swell, in 38 instances out of 48 was less when an additive was used. Although no definite pattern of behavior can be established, some indication of the beneficial effect of the additives can be obtained by study of the data shown. Probably a better comparison of the effectiveness of the additives in reducing volumetric swell can be obtained from table 5. The data show that all three additives were of some benefit in reducing volumetric swell and that additive A was the most effective.

Table 5.—Comparison of the effectiveness of additives in reducing moisture absorption and volumetric swell

	Additives			
	None	A	B	C
RANGE AND AVERAGE VALUES				
Moisture absorption:	<i>Pct.</i>	<i>Pct.</i>	<i>Pct.</i>	<i>Pct.</i>
Minimum.....	1.8	2.1	2.3	1.9
Maximum.....	4.2	2.7	3.1	3.2
Average.....	3.1	2.4	2.8	2.5
Volumetric swell:				
Minimum.....	.6	.3	.3	.4
Maximum.....	3.2	1.0	1.7	1.8
Average.....	1.5	.6	1.0	1.2
FREQUENCY OF OCCURRENCE IN 16 TESTS				
Moisture absorption (percent):				
2.0 or less.....	1	0	0	2
2.1-2.5.....	2	10	4	6
2.6-3.0.....	5	6	9	7
3.1-3.5.....	4	0	3	1
More than 3.5.....	4	0	0	0
Volumetric swell (percent):				
0.5 or less.....	0	5	1	1
0.6-1.0.....	5	11	9	4
1.1-1.5.....	4	0	5	7
1.6-2.0.....	4	0	1	4
More than 2.0.....	3	0	0	0

# Properties of Concrete Containing a Blend of Portland Cement and Air- Entraining Ground Blast-Furnace Slag

BY THE PHYSICAL RESEARCH BRANCH  
BUREAU OF PUBLIC ROADS

Reported by WILLIAM E. GRIEB and GEORGE WERNER  
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*The results of the tests reported in this article indicate that finely ground blast-furnace slag containing an air-entraining admixture can be used advantageously in concrete as a blend with non-air-entraining portland cement. The durability of concrete containing this blend was equal or superior to that of corresponding concretes made with either air-entraining or non-air-entraining portland cements used alone or blended with natural cement. Similarly, favorable strength performance was observed except in comparison to concrete made with unblended non-air-entraining portland cement.*

FINELY ground water-granulated blast-furnace slag containing a commercial air-entraining agent has been developed recently for use as a blend with portland cement in the same ratio as natural cement is commonly blended with portland cement (one bag of natural cement plus six bags of portland cement in a seven-bag batch). This report presents the results of a series of tests to determine the effect of using a blend of a type II non-air-entraining portland cement and the finely ground blast-furnace slag containing an air-entraining admixture on the strength and durability of paving concrete. For comparison, similar tests also were made on concrete containing the unblended type II portland cement, the unblended air-entraining counterpart of that cement (type IIA), and blends of each of these portland cements and natural cement. Proportions were similar to those which have been generally employed for many years in cases where blends of portland and natural cement have been used in pavement construction.

Many of the Northeastern States have been using, for the past 20 years or so, a blend of portland and natural cement in the construction of concrete pavements. The practice has been established largely on the basis of field experience which has indicated that the replacement of one bag of portland cement by one bag of natural cement in the average pavement mix will improve the resistance of the concrete to weathering without significant loss in strength. Natural cements as used in the Northeastern area are of two types: a natural cement to which there has been added

a very small amount of beef tallow (0.05 to 0.10 percent), which is designated as type N by State specifications, and a natural cement which has been plasticised by adding a relatively large quantity (0.20 to 0.30 percent) of beef tallow. The former type only was used in this investigation.

## Conclusions

The results of this series of tests indicate that ground blast-furnace slag containing an air-entraining admixture can be used advantageously in concrete as a blend with non-air-entraining portland cement in the proportions of approximately 14 percent ground slag to 86 percent portland cement, by volume.

For the materials used in these tests, the durability of concrete containing this blend (as measured by laboratory freezing and thawing tests) was far superior to that of the corresponding concrete containing either the type II non-air-entraining portland cement or the blend of that cement with the type N natural cement and was as good as that of the concrete containing the type IIA air-entraining portland cement and the blend of that cement with natural cement. The strength of concrete containing the blend of the type II cement and ground slag, while not as high as that of the concrete containing the type II portland cement, was, on the average, somewhat higher than the concrete containing the blend of type II portland and natural cement. Furthermore, the strength of this combination was, in all but one case, higher than that of the concrete containing the type IIA portland cement, either unblended or blended with natural cement.

## Materials Used

The following materials were used in this investigation:

- Portland cement, ASTM type II.
- Air-entraining portland cement, ASTM type IIA, from the same plant.
- Natural cement, New York State type N.<sup>1</sup>
- Ground water-granulated blast-furnace

<sup>1</sup> See Specifications, State of New York Department of Public Works, Division of Construction, dated Jan. 2, 1947, p. 73.

slag containing 0.090 percent of an air-entraining admixture.

Siliceous sand from White Marsh, Md.; fineness modulus, 2.80; specific gravity, 2.64 (bulk dry basis).

Crushed limestone from Riverton, Va.; specific gravity, 2.78 (bulk dry basis); 1½-inch maximum size for strength specimens and 1-inch for durability specimens.

The results of chemical and physical tests on the cements are shown in table 1. The portland cements conformed to the specification requirements of the applicable ASTM specifications in all respects. The natural cement met the requirements of ASTM specification C 10-37 for soundness, time of setting, and tensile strength at 28 days. It failed to meet the minimum tensile strength of 75 p.s.i. at 7 days required by this specification. The ground blast-furnace slag met the chemical requirements of ASTM specification C 205-48 T (portland blast-furnace slag cement) for the slag constituent of this cement. It also met the 7- and 28-day tensile strength requirements of this specification but failed to attain final set at 10 hours, as required by the specification.

## Combinations and Proportions

The various cement combinations and proportions used in these tests, as well as the results of tests on the plastic concrete, are shown in table 2. Mixes 1-5 were used in the strength-test specimens; mixes 6-10 were used in the specimens tested for durability.

For the mixes containing unblended portland cement (Nos. 1, 4, 6, and 9) the proportions were adjusted to give a cement content of 6.4 bags per cubic yard, which is approximately the cement factor used in concrete paving in most of the Northeastern States. In the case of the mixes with blended cement (Nos. 2, 3, 5, 7, 8, and 10) replacements for a portion of the portland cement by natural cement or ground slag were made on the basis of a blend containing 86 percent portland cement and 14 percent natural cement or ground slag, by volume. This is approximately the ratio used in practice where it is customary to substitute one 80-pound (1 cubic

**Table 1.—Chemical composition and physical properties of cements and ground slag**

	Portland cement		Natural cement	Gr'nd slag
	Non-air-entraining	Air-entraining		
<b>Chemical composition (in percent):</b>				
Silicon dioxide.....	21.2	21.2	23.6	33.4
Aluminum oxide.....	5.7	5.7	5.4	10.7
Ferrous oxide.....	---	---	---	1.2
Ferric oxide.....	4.5	4.5	4.4	---
Calcium oxide.....	63.0	63.0	33.1	46.4
Magnesium oxide.....	2.1	2.2	20.9	5.3
Sulfur trioxide.....	1.5	1.5	1.5	.57
Manganic oxide.....	---	---	---	.44
Sulfide sulfur.....	---	---	---	1.27
Total sulfur.....	---	---	---	1.50
Sodium oxide.....	.16	.16	.28	---
Potassium oxide.....	.62	.64	1.52	---
Loss on ignition.....	1.1	1.0	9.2	11.70
Insoluble residue.....	.12	.16	14.5	.21
Chloroform-soluble material.....	.005	.008	.032	.0004
Tricalcium silicate.....	46	46	---	---
Dicalcium silicate.....	26	26	---	---
Tricalcium aluminate.....	8	8	---	---
Tetracalcium aluminoferrite.....	14	14	---	---
Calcium sulfate.....	2.5	2.5	---	---
<b>Physical properties:</b>				
Apparent specific gravity.....	3.17	3.19	3.05	2.94
Specific surface cm. <sup>2</sup> /gm.....	1,740	1,640	---	---
Autoclave expansion percent.....	.02	.04	---	---
Normal consistency percent.....	23.0	23.0	31.2	29.2
Time of set (Gillmore):				
Initial.....hours.....	5.2	5.0	2.3	23.5
Final.....do.....	7.2	7.5	4.1	210+
Tensile strength, 1:3 mortar:				
At 7 days p.s.i.....	375	370	40	380
At 28 days do.....	490	455	190	450
Soundness (pat test).....	---	---	OK	OK
Air content (mortar test) percent.....	10.7	17.0	13.2	15.7

<sup>1</sup> Gain in weight. <sup>2</sup> Vicat test. <sup>3</sup> 1:2 mortar.  
<sup>4</sup> 14 percent natural cement; 86 percent portland cement (by weight).  
<sup>5</sup> 12.5 percent ground slag; 87.5 percent portland cement (by weight).

foot) bag of natural cement or ground slag for one 94-pound (1 cubic foot) bag of portland cement in a seven-bag batch. The relative quantities used were such as to maintain the same total volume of cementitious material per unit volume of concrete as was used in the unblended mixes.

**Table 2.—Cement combinations, proportions, and tests of plastic concrete**

Mix No.	Combinations used, and quantities per cubic yard of concrete							Tests on plastic concrete		
	Portland cement		Natural cement	Ground slag	Aggregate		Water	Slump	Weight	Air content (gravimetric)
	Type II	Type IIA			Fine	Coarse <sup>1</sup>				
For strength tests:	Lb.	Lb.	Lb.	Lb.	Lb.	Lb.	Lb.	In.	Lb./cu. ft.	Pct.
1 <sup>2</sup> .....	602	0	0	0	1,203	2,035	271	3.0	152.2	2.5
2.....	517	0	72	0	1,203	2,035	267	2.7	151.6	2.8
3.....	517	0	0	72	1,107	2,035	253	2.8	148.2	5.4
4.....	0	602	0	0	1,107	2,035	254	2.8	148.3	5.6
5.....	0	517	72	0	1,107	2,035	260	2.8	147.4	4.4
For durability tests:										
6 <sup>2</sup> .....	602	0	0	0	1,210	1,940	287	2.4	150.3	2.3
7.....	517	0	72	0	1,210	1,940	285	2.6	149.7	2.7
8.....	517	0	0	72	1,115	1,940	268	2.8	145.9	5.7
9.....	0	602	0	0	1,115	1,940	267	3.0	146.2	6.1
10.....	0	517	72	0	1,115	1,940	273	2.6	146.9	4.9

<sup>1</sup> Maximum aggregate size was 1½ inches for strength tests and 1 inch for durability tests.  
<sup>2</sup> Base or reference mixes.

**Table 3.—Results of strength tests**

Mix No.	Cement combinations			Air content (gravimetric)	Strength tests of concrete						
	Portland cement	Blending material	Pct.		Flexure: Modulus of rupture <sup>1</sup> at—			Compression (beams <sup>2</sup> ) at—			Compression (cylinders <sup>3</sup> ) at 14 days
					14 days	90 days	365 days	15 days	91 days	366 days	
1	Type II.....	None.....	2.5	P. s. i. 840	P. s. i. 900	P. s. i. 940	P. s. i. 6,140	P. s. i. 6,560	P. s. i. 8,750	P. s. i. 5,140	
2	Type II.....	Natural cement.....	2.8	805	935	845	5,400	6,450	7,200	4,600	
3	Type II.....	Ground slag.....	5.4	830	915	865	5,550	6,280	7,800	4,490	
4	Type IIA.....	None.....	5.6	770	840	785	5,120	6,160	6,720	4,640	
5	Type IIA.....	Natural cement.....	4.4	725	890	770	4,590	5,500	7,060	4,280	

<sup>1</sup> Tests made on 6- by 6- by 21-inch beams, tested in accordance with ASTM standard method C 78-49, with sides as molded in tension. Loaded at third points of an 18-inch span. Each value average of two or three tests.

<sup>2</sup> Tests made on portions of beams remaining after flexure test, in accordance with ASTM standard method C 116-49. Each value average of four or six tests.

<sup>3</sup> Tests made on 6- by 12-inch cylinders in accordance with ASTM standard method C 39-49. Each value average of three tests.

For the base or reference mixes (Nos. 1 and 6) a ratio of fine to coarse aggregate was established to approximate an average concrete pavement mix, using crushed stone as coarse aggregate. For the materials used in these tests, the base mixes entrained about 2.5 percent air. It was found that the blending of the natural cement with the non-air-entraining portland cement (mixes No. 2 and 7) resulted in the entrainment of very little additional air. Therefore, no adjustment in sand content was made in the case of these mixes. In all the other combinations, however, a substantial increase in air content was noted so that, in each of these cases, it was necessary to reduce the fine aggregate content sufficiently to maintain the same total quantity of cementitious material per cubic yard of concrete.

All concretes had a slump ranging from 2.4 to 3.0 inches. For this consistency the air-entrained concrete required approximately 2 gallons of water less per cubic yard of concrete than was required for the non-air-entrained concrete.

**Test Procedures**

Because of the extensive use of blends of natural and portland cements in paving concrete in several of the Northeastern States, as well as in certain other sections of the country, it was decided to make the primary strength study an investigation of the flexural strength of the concrete and to determine the com-

pressive strength by means of tests on modified cubes from the broken halves of the beams remaining after the flexural tests. However, as a check on the modified cube tests, 6- by 12-inch cylinders were made for test at 14 days only. The flexural test specimens were 6- by 6- by 21-inch beams, and were tested in accordance with ASTM standard method C 78-49. The modified cubes were 6 by 6 inches in cross section and were tested in accordance with ASTM method C 116-49. Flexure specimens were tested after 14, 90, and 365 days in moist storage. The broken beams were tested as compression specimens on the following days. The results of the strength tests are shown in table 3.

Specimens for durability testing (as determined by freezing and thawing in water) were 3- by 4- by 16-inch beams. Cement contents were the same as for the strength tests. However, the maximum size of the coarse aggregate was 1 inch instead of 1½ inches, with a corresponding change in the ratio of fine to coarse aggregate necessitated by the smaller maximum size (see table 2, mix Nos. 6-10).

Two groups of specimens were made. In the first group, 3- by 4- by 16-inch beams were sawed from 4- by 16- by 24-inch slabs in such a way as to provide seven beams, each having two parallel sawed surfaces. Prior to sawing, the slabs were cured under wet burlap for 7 days, followed by 28 days storage in laboratory air. After sawing, four of the test beams from each slab were placed in moist air storage to be tested as control specimens. The other three were immersed in water for 48 hours after which the freezing and thawing test was started. In the second group, individual 3- by 4- by 16-inch beams were molded in three-gang molds from the same batch of concrete used for molding the slabs. These beams were moist-cured continuously until the start of the freezing and thawing test. No individual beams for control tests were made. All specimens were frozen and thawed in water at a temperature range from 70° to -20° F. A rather slow cycle was used, approximately 7 hours being required to reduce the temperature of the concrete from 70° to -20° F. One complete cycle required 24 hours, the specimens being placed in the freezing chamber at about 4 p. m., remaining there until about 9 a. m. the following morning,

and then being placed in the thawing tank until 4 p. m.

The natural frequency of vibration of each specimen, expressed as  $N^2$  (natural frequency of vibration, squared), was determined sonically at 70° F. at the start of the test and at periodic intervals thereafter. Alternate freezing and thawing was continued for 200 cycles or until the specimens showed a drop in  $N^2$  of 40 percent, whichever occurred first. The reduction in  $N^2$  at periodic intervals, expressed as percentages of the corresponding values at zero cycles, is shown in table 4. Also shown are the durability factors and the reductions in flexural strength expressed as percentages of the strength of moist-cured specimens tested at the same time. The durability factors were calculated from the following formula:

$$DF = \frac{(100 - R)N}{200}$$

Where

$DF$  = durability factor.

$R$  = reduction in  $N^2$  (value will be 40 percent or less).

$N$  = number of cycles at which  $R$  was reduced 40 percent, or 200, whichever occurred first.

This formula is the same as that shown in ASTM tentative specification C 260-50 T, except that values for  $N^2$  were used in place of the dynamic modulus of elasticity and calculations were based on a reduction in  $N^2$  of 40 percent instead of 30 percent.

### Discussion of Results

The results of the tests are summarized in table 5 which shows, for each test age, the relative strength and durability of each combination as compared to the base mix (concrete containing the unblended type II portland cement). The trends revealed by these data will be discussed briefly, first with respect to strength and second with respect to durability.

Reference to table 3 shows that, except in two cases, the base mix (No. 1) developed higher strengths both in flexure and compression than any of the other combinations. Reductions in strength (table 5) varied from an average of about 7 percent for the blend of type II portland cement and ground slag (No. 3) to an average of about 16 percent in the case of the blend of type IIA portland cement and natural cement (No. 5). The blend of type II portland cement and natural cement (No. 2) and the unblended type IIA portland cement (No. 4) showed average percentage reductions intermediate between these extremes. Reductions in strength shown for mix Nos. 4 and 5 are in line with what would be expected due to the relatively high air content of these combinations. However, mix No. 3, with next to the highest air content, showed the smallest average reduction in strength—actually less than mix No. 2, which had an air content only 0.3 percent higher than the base mix.

It will be noted from table 3 that all of the combinations except the base mix showed retrogression in flexural strength at 1 year as compared to the strength at 90 days. This

Table 4.—Results of durability tests

Mix No.	Cement combination		Type of specimen	Air content (gravimetric)	Reduction in $N^2$ after freezing and thawing for -- cycles										Durability factor	Reduction in flexural strength <sup>1</sup>
	Portland cement	Blending material			10	25	50	75	100	125	150	175	200			
					Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.		
6	Type II	None	Molded	2.3	9	17	28	30	43	---	---	---	---	---	29	54 (120)
			Sawed	2.3	9	13	23	22	28	239	---	---	---	---	36	
7	Type II	Natural cement	Molded	2.7	9	18	22	23	30	240	---	---	---	---	36	53 (75)
			Sawed	2.7	8	14	31	37	---	---	---	---	---	---	24	
8	Type II	Ground slag	Molded	5.7	6	13	14	16	20	26	33	35	39	61	52 (200)	
			Sawed	5.7	6	9	14	15	20	25	29	30	35	65		
9	Type IIA	None	Molded	6.1	7	13	18	18	23	30	39	40	41	60	46 (200)	
			Sawed	6.1	3	5	12	12	15	20	26	27	32	68		
10	Type IIA	Natural cement	Molded	4.9	8	14	18	18	21	28	35	35	36	64	54 (200)	
			Sawed	4.9	6	8	12	14	15	22	27	30	33	67		

<sup>1</sup> Reduction in flexural strength after alternate freezing and thawing expressed as a percentage of the strength of corresponding control specimens stored continuously in moist air and tested at the same time. Values in parentheses are number of cycles at which test was made.

<sup>2</sup> These final readings were at 120 cycles.

is somewhat surprising, especially in view of the fact that a type II cement was used as the portland cement constituent in all of the blends as well as in the unblended air-entrained portland cement concrete. This tendency also has been noted in another current investigation of the Bureau where the effect of air-entraining admixtures on strength is being investigated. However, it was not evident in the study of age-strength relations for air-entrained concrete recently reported in PUBLIC ROADS,<sup>2</sup> nor was it evident in the study of various natural cement blends published in PUBLIC ROADS in 1947.<sup>3</sup>

In contrast with the flexural strength results, the compressive strength of all combinations increased between 91 and 366 days although the rate of increase was somewhat less for the blends and the unblended air-entrained concrete than for the base mix. From table 3, it will also be noted that the results of the 14-day compression tests of 6-by 12-inch cylinders, while considerably lower, parallel in general the 15-day tests on the modified cubes.

The three combinations which contained substantial amounts of entrained air (Nos. 8, 9, and 10) were much more resistant to alternate freezing and thawing than the two mixes

<sup>2</sup> Age-strength relations for air-entrained concrete, by F. H. Jackson. PUBLIC ROADS, vol. 27, No. 2, June 1952.

<sup>3</sup> The effect on properties of concrete of natural and portland cement blends, by A. G. Timms, W. E. Grieb, and Geo. Werner. PUBLIC ROADS, vol. 25, No. 2, Dec. 1947.

(Nos. 6 and 7) with relatively low air content, as may be seen in tables 4 and 5. The average of the durability factors for the three mixes of high air content was about 65 as compared to an average value of about 30 for the two mixes of low air content. Except for mix No. 7, test specimens sawed from the 4- by 16- by 24-inch slabs showed higher resistance than the corresponding molded beams. This was due, no doubt, to the 28-day drying period introduced during the curing of the slabs. The molded beams were continuously moist cured until tested. The low resistance shown by the combination containing the non-air-entraining portland cement and natural cement (No. 7) is in line with the results which were reported for a similar combination in 1947.<sup>4</sup> However, the reversal in trend with respect to the comparative durability of the sawed vs. the molded beams shown by this combination has not been explained.

Referring again to table 4, it will be noted that the reduction in flexural strength after freezing and thawing parallels, in general, the reduction in  $N^2$ , thus verifying the assumption that the decrease in the natural frequency of vibration of a concrete specimen as the result of alternate freezing and thawing is a measure of its loss in strength. It should be noted, however, that no direct comparison between loss in flexural strength and dura-

<sup>4</sup> See footnote 3.

Table 5.—Summary of test results

Mix No.	Cement combination		Air content (gravimetric)	Decrease in strength as compared to strength of base mix (No. 1)								Change in durability factor				
	Portland cement	Blending material		In flexure, after—				In compression, after—				Average <sup>1</sup>	Sawed beams	Molded beams	Average	
				14 days	90 days	365 days	14 days	15 days	91 days	366 days						
				Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.	Pct.					
1	Type II	None	2.5	---	---	---	---	---	---	---	---	---	---	---	---	---
2	Type II	Natural cement	2.8	4	+4	10	11	12	12	18	9	---	---	---	---	---
3	Type II	Ground slag	5.4	1	+1	8	13	10	4	11	7	---	---	---	---	---
4	Type IIA	None	5.6	8	7	16	10	17	6	23	12	---	---	---	---	---
5	Type IIA	Natural cement	4.4	14	1	18	17	25	16	19	16	---	---	---	---	---
6	Type II	None	2.3	---	---	---	---	---	---	---	---	---	---	---	---	---
7	Type II	Natural cement	2.7	---	---	---	---	---	---	---	---	---	---	-12	+7	-2
8	Type II	Ground slag	5.7	---	---	---	---	---	---	---	---	---	---	+29	+32	+30
9	Type IIA	None	6.1	---	---	---	---	---	---	---	---	---	---	+32	+31	+32
10	Type IIA	Natural cement	4.9	---	---	---	---	---	---	---	---	---	---	+31	+35	+33

<sup>1</sup> Average of flexure and compression values.

bility factor is possible due to the variable number of cycles of freezing and thawing at which the strength tests were made. On the other hand, comparison can be made between the loss in flexural strength and the loss in  $N^2$

after the same number of cycles of freezing and thawing. For example, the 54-percent loss in flexural strength for the base mix (No. 6) at 120 cycles can be compared to the drop in  $N^2$  of 39 percent at the end of the same

number of cycles. For the several combinations, the ratio of the loss in strength to the drop in  $N^2$  varied from 1.4 to 1.6. Previous investigations have shown that this ratio is usually 1.2 or greater.

# A Comparison of Rapid Methods for the Determination of Liquid Limits of Soils

BY THE PHYSICAL RESEARCH BRANCH  
BUREAU OF PUBLIC ROADS

Reported by FRANK R. OLMSTEAD  
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Engineers responsible for the routine testing of large numbers of soil samples for highway construction and maintenance need rapid methods for testing. In this study, two simplified methods of determining the liquid limit of soil are compared with the standard method to indicate their reliability and accuracy. Both are "one-run" methods, that is, they require a single determination of moisture and number of blows for groove closure to calculate the liquid limit.

The methods discussed—the chart method developed by the Washington State Department of Highways and a modification of the chart method involving use of a slide rule—have been compared for a wide range of soils found in many of the States. Both of these methods are rapid and reduce the time normally required for testing by as much as 30 to 70 percent. They were found to have good reproducibility and to have sufficient accuracy, within limits, to be acceptable as alternate test procedures for the standard method used for the determination of the liquid limit soil constant.

THE lower liquid limit, developed by Albert Atterberg, was suggested in 1926 as one of the soil constants that could be used to evaluate a soil as a road-building material.<sup>1</sup> This soil constant is now widely used by highway and airport engineers to evaluate soils for use as subgrade, base course, and embankment material. High liquid limits usually indicate heavy clays and low values indicate friable soils, silts, and clayey sands.

The lower liquid limit, now known as the liquid limit, is defined as the percentage of moisture, based on the dry weight of soil, at which a soil will just begin to flow when jarred slightly. According to this definition, soils at their liquid limit have a small but definite shear resistance which can be overcome by the application of very little force. The cohesion of the soil-water mixture at the liquid limit is practically zero.

The determination of this soil constant as originally proposed was made by a hand method<sup>1</sup> in which the operator adjusted the moisture content by trial and error until exactly 10 blows closed a standard width groove made in the soil sample. The moisture content for this soil sample was taken as the liquid limit of the soil. This method required considerable skill and judgment by the operator to obtain duplicate test results.

As soil testing became more widely used, the need for a more practical test procedure became apparent and a mechanical device<sup>2</sup>

was developed which eliminated personal judgment in estimating the proper intensity of the blows to cause closure of the groove. Although the liquid limit machine was found to give good reproducibility of results and has been adopted as the AASHTO standard test method,<sup>3</sup> it requires more time than the hand method.

The standard method requires that three random trials be made, each at a different moisture content and number of blows; that the percentage of moisture and number of blows be plotted on semilog paper and a flow curve drawn through the points; and that the liquid limit be taken as the moisture content corresponding to the point where the flow curve intersects the 25-blow line. This procedure has less probable error than a single determination by the hand method, and the machine can be used by operators of less skill; but the additional trials, weighings, and computations require considerably more time than that required by skilled operators familiar with the use of the one-point hand method.

It is reasonable to expect that engineers responsible for routine testing of soils would be reluctant to accept a longer test procedure without carefully examining the method to determine if modifications could be made which would permit more rapid testing of soils.

One of the first modifications to be tried was patterned after the hand method, by using the mechanical liquid-limit device to furnish

the standard force and adjusting the soil-water consistency by trial and error until exactly 25 blows of the machine closed the standard width groove.

This modification of the machine method, usually referred to as the moisture adjustment method, can be developed into an accurate, rapid method by skilled operators if calibrated with the standard method, and it can be used effectively to reduce the time required for the determination of the liquid limit of a soil. However, it is difficult for inexperienced or less skilled operators to adjust the consistency of the soil-water mixture to obtain groove closure at exactly 25 blows.

## Chart Method

A rapid method for determining the liquid limit has been devised by Cooper and Johnson of the Washington State Department of Highways<sup>4</sup> for the routine testing of soils in their laboratory. It is called the chart method and is based upon the relation between the slopes of flow curves obtained for soils with different liquid limit test values. Cooper and Johnson observed, in their initial study of soils taken from various parts of the State of Washington, that the flow index<sup>5</sup> computed from the flow curves obtained by the AASHTO standard method of test increased uniformly with the liquid limit of the soils. From these data, six typical flow curves, A-F shown in figure 1, were developed.

Cooper and Johnson established the validity of these six typical curves by plotting the number of blows and moisture contents for single points representing random trial run on each of 27 different soils. In this series of tests, the number of blows for groove closure was varied within a range of 7 to 60. The liquid limit was determined for each soil by drawing a line through the plotted point and parallel to the nearest typical flow curve and recording, as the liquid limit, the moisture content at the intersection of this line with the 25-blow line. An example is shown by dashed lines in figure 1.

<sup>1</sup> A rapid method of determining the liquid limit of soils, by J. H. Cooper and K. A. Johnson. Materials Laboratory Report No. 83, Washington State Department of Highway March 1950.

<sup>5</sup> Flow index is the range in moisture content represented by the number of blows or shocks in one cycle of the logarithmic scale of a flow curve.

<sup>1</sup> Adaptation of Atterberg plasticity tests for subgrade soils, by A. M. Wintermyer. PUBLIC ROADS, vol. 7, No. 6, Aug. 1926.

<sup>2</sup> Research on the Atterberg limits of soils, by Arthur Casagrande. PUBLIC ROADS, vol. 13, No. 8, Oct. 1932.

<sup>3</sup> Standard method of determining the liquid limit of soils, AASHTO designation T 89-49. Standard Specifications for Highway Materials and Methods of Sampling and Testing, part II, 6th ed., p. 228. Published by the American Association of State Highway Officials. 1950.

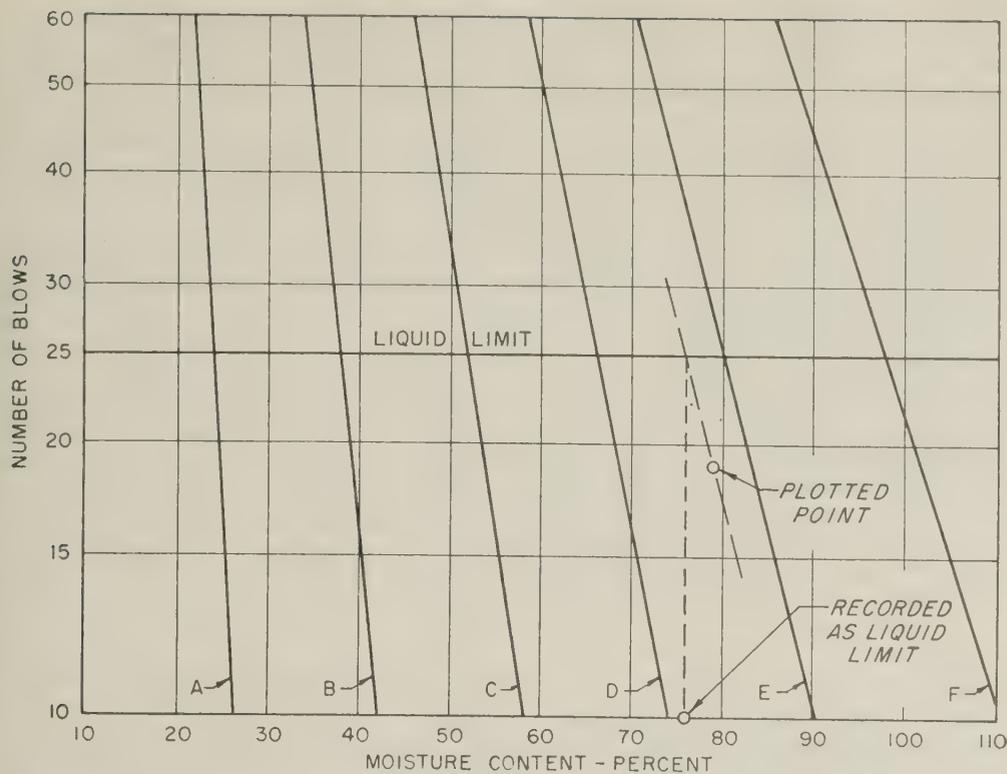


Figure 1.—Relation between slope of typical flow curves and liquid limit.

The values thus obtained from the series of tests were compared with the liquid limits determined by the standard method and good agreement was found between the computed values obtained by this chart method and those determined by the standard test procedure.

Cooper and Johnson made a further simplification of the chart method by subdividing their original six-curve chart to show typical flow curves for each percentage of moisture, so that any point selected from a single moisture content and corresponding number of blows for groove closure could be projected visually to the 25-blow line to obtain the liquid limit.

This improved chart, shown in figure 2, was checked by Cooper and Johnson by using 84 duplicate tests of 73 soil samples. In each case, the liquid limit was determined by the

moisture adjustment method and by the chart method. This comparison between the two methods of test showed that discrepancies ranged from 0 to 1.8 percentage points. Because of the low frequency of errors in excess of 1.0 percentage point, however, they considered the accuracy of the chart method adequate for soil classification purposes. They pointed out that two separate closures of the soil-water mixture should be observed to check its consistency before selecting a moisture sample for the determination of the liquid limit by this revised chart method. It was their opinion that the operator should, after completing the second closure, be sufficiently familiar with the flow characteristics of the material to detect any erroneous results.

Although this method is used in their routine identification and classification of soils, Cooper and Johnson recommend that it

should not be used for the acceptance or rejection of materials which have borderline values of liquid limit or plasticity index. In these cases, they recommend the use of the AASHO standard mechanical method (designation T 89-49).

The results of a statistical analysis of the basic data used by Cooper and Johnson in the preparation of their original and revised chart methods are shown in table 1. The analysis shows that reduction from the 7-60 to the 15-40 range in the number of blows for groove closure increased the accuracy of the chart method. The maximum deviation in the revised chart method for the 15-40 blow range was 2 percentage points. The deviation was less than 1 point for 88 percent of the tests in the 15-40 blow range, as compared to 70 percent of the tests in the 7-60 blow range.

### Chart Method Investigated

Since only soils from the State of Washington had been studied by Cooper and Johnson, and the moisture adjustment method was the yardstick of comparison, the Bureau of Public Roads widened the scope of the investigation by checking the new method against the AASHO standard method (designation T 89-49) for 364 soil samples selected to represent soils from all sections of the United States. Figure 3 shows the areas from which these samples were obtained. The soils selected had liquid limits ranging from 15 to 104.

The soil test data for checking the accuracy of the chart method were taken from laboratory records and two points with the highest and lowest number of blows were selected from each standard flow curve for each of the 364 soil samples. The moisture contents corresponding to these two numbers of blows for these two points were used for calculating the liquid limit from the revised chart (fig. 2).

To compare the accuracy of these results with those obtained by the standard test method, the data were subdivided according to their liquid limit values into six groups. The first group was terminated at 25 since that is the maximum liquid limit allowed for base-course materials by the AASHO standard specifications, and it is also the upper limit for A-1 soils in the AASHO system of soil classification.<sup>6</sup>

The second group was terminated at 40, the maximum liquid limit for A-4 and A-6 soil groups in the AASHO system of soil classification. The liquid limit values above 40 were arbitrarily subdivided into four 15-point ranges. A tabulation of the deviations of 759 liquid limit values calculated by the chart method from those determined by the AASHO standard mechanical method is shown in table 2. Since the groups do not contain the same number of tests, the frequencies of the deviations, column 4, are expressed in the form of percentages to facilitate comparisons.

<sup>6</sup> Standard recommended practice for the classification of soils and soil-aggregate mixtures for highway construction purposes, AASHO designation M 145-49. Standard Specifications for Highway Materials and Methods of Sampling and Testing, part I, 6th ed., p. 29. Published by the American Association of State Highway Officials, 1950.

Table 1.—Comparison of deviations in values of liquid limit obtained by the Cooper and Johnson chart methods from those obtained by standard tests<sup>1</sup>

Range of deviations <sup>2</sup> (percentage points)	Deviation from standard method values of—					
	Six-curve chart method values (groove closure in 7-60 blows)			Multicurve chart method values (groove closure in 15-40 blows)		
	Number of tests deviating in indicated range	Percentage of total number of tests	Cumulative percentage	Number of tests deviating in indicated range	Percentage of total number of tests	Cumulative percentage
0.....	4	6	6	8	9	9
0.1-0.5.....	28	38	44	41	48	57
0.6-1.0.....	19	26	70	28	31	88
1.1-1.5.....	6	8	78	7	8	96
1.6-2.0.....	6	8	86	3	4	100
2.1-2.5.....	6	8	94	-----	-----	-----
2.6-3.0.....	1	1	95	-----	-----	-----
3.1-3.5.....	2	3	98	-----	-----	-----
3.6-4.0.....	0	0	98	-----	-----	-----
over 4.0.....	2	2	100	-----	-----	-----
Total.....	74	100	-----	85	100	-----

<sup>1</sup> Duplicate samples tested, one at random blows and the other by the standard test procedure used in the Washington State Department of Highways laboratory.

<sup>2</sup> Deviations  $\pm$  from the value of liquid limit determined by moisture adjustment method.

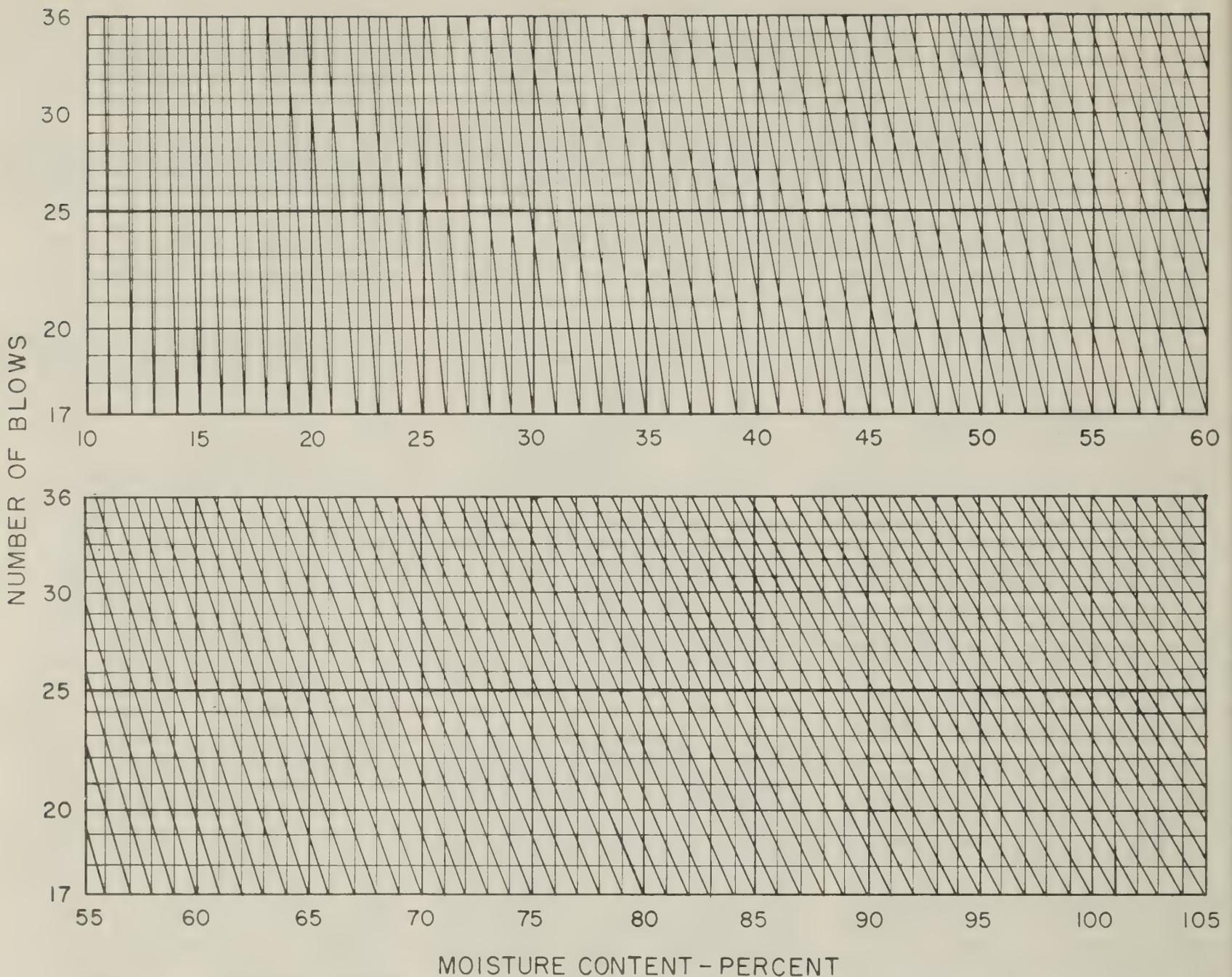


Figure 2.—Multicurve chart developed by Washington State Department of Highways for calculation of the liquid limit.

These data, obtained for 364 soil samples tested by the Bureau of Public Roads, show a range in deviations from the standard values of 0 to about 2 percentage points. This range of deviations compares quite favorably with the range 0 to 1.8 reported by Cooper and Johnson for test data obtained for 73 soil samples taken from the State of Washington.

#### Slide-Rule Method Developed

The investigation of the accuracy of the chart method for calculation of the liquid limit suggested the possibility of eliminating the use of the chart by the development of a special scale which could be inscribed on a slide rule.

This was accomplished by deriving an equation for the family of typical curves used by Cooper and Johnson in the preparation of their chart. A study of the typical curves in figure 1 indicated a point of convergence near the zero moisture content axis, and while it would be possible to derive an equation for such a family of curves, the problem was simplified by arbitrarily moving the point of

convergence to the zero axis. This point is so far from the 25-blow line that the consequent changes in the slopes of the flow curves are negligible within the 17-36 blow range used. The formula derived for this new family of curves is:

$$LL = \frac{W}{1.419 - 0.3 \log S}$$

where

$LL$  = liquid limit.

$S$  = number of blows (or shocks) required to close the groove.

$W$  = percentage of moisture contained by the soil when  $S$  blows close the groove.

The use of this formula is facilitated by computing a table of the denominator ( $1.419 - 0.3 \log S$ ) for different values of  $S$ , as shown in table 3. The liquid limit for any moisture content at groove closure between 15 and 40 blows is obtained by dividing the percentage of moisture by the value of the denominator in table 3 corresponding to the number of blows required to close the groove.

A more convenient method for using the formula and the data in table 3 is to inscribe a special scale in the space below the B scale of a slide rule to indicate the number of blows required for groove closure corresponding to the values of the denominator of the formula. Figure 4A shows a section of a 20-inch poly-phase slide rule with this special scale inscribed for the number of blows between 15 and 40. A study of this special scale with respect to the normal B scale of the slide rule shows that it is constructed by making a mark below the B scale of the slide rule for each value of the denominator of the formula. For example, the 20-blow mark on the special scale corresponds to a B scale reading of 1.029, the 25-blow mark to a value of 1.000 (the midpoint of the B scale), the 30-blow mark to a reading of 0.976, etc.

The calculation of the liquid limit by the slide-rule method is easily made by setting the indicator line over the percentage of moisture found in the soil sample on the A scale of the slide rule and adjusting the slide so that the corresponding number of blows

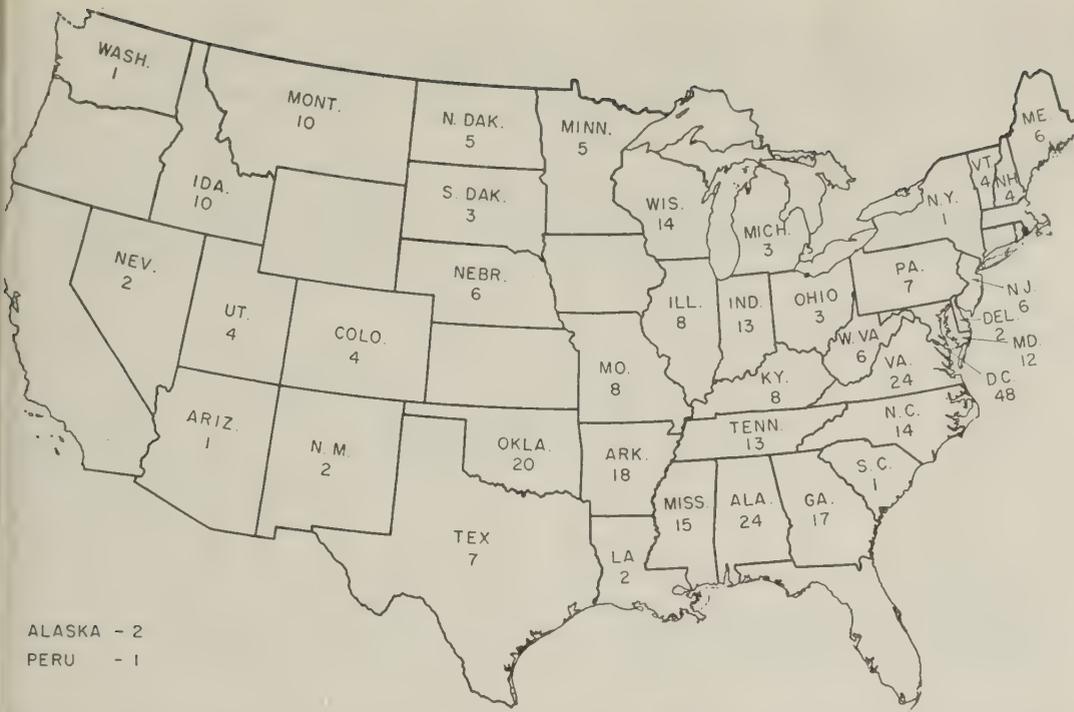


Figure 3.—Number and distribution of soil samples used in testing the relation between the one-point liquid limit tests and the AASHO standard method.

on the special scale coincides with the indicator line. With this setting of the slide rule, the liquid limit is read on the A scale above the end index of the B scale, or on the A scale directly above the 25-blow mark on the special scale.

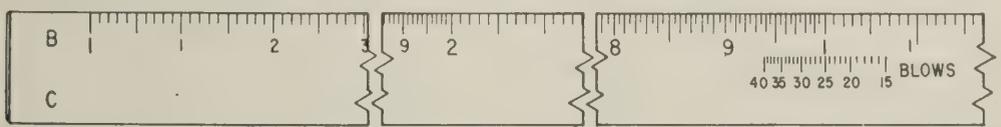
In the example shown in figure 4B, 20 blows were required to close the groove and the corresponding moisture content was 21.4 percent. The calculated liquid limit, shown on the A scale above the end index of the B scale, is 20.8.

**Comparison of Two Methods**

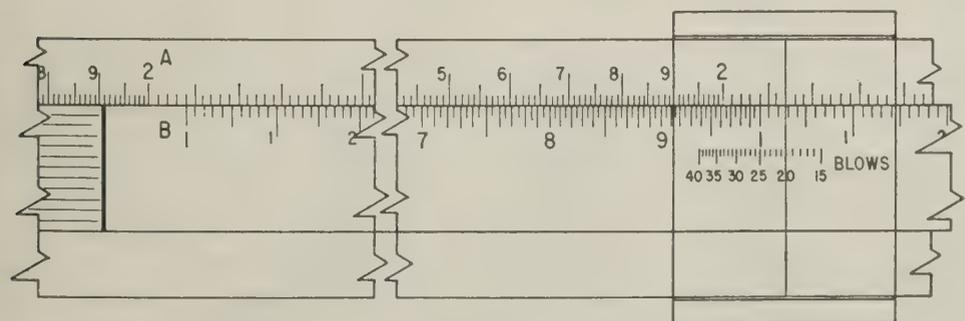
The reproducibility of the slide-rule method was checked against the standard method for the same data that were used to check the revised chart method developed by Cooper and Johnson (see table 2, fourth column). The percentage frequency for the same devia-

tions is shown in the last column of table 2, so that comparisons can be made with the chart method. Figure 5 provides a graphic comparison of the accuracy of the slide-rule method and the revised chart method for the ranges of liquid limits selected for this study. The number of tests deviating by  $\pm 0$ ,  $\pm 1$ , and  $\pm 2$  percentage points from the liquid limits determined by the AASHO standard method is expressed on a percentage basis to facilitate the comparison of the accuracy of the two methods.

It is shown by these comparisons that, within the range of liquid limits most generally encountered, the chart and slide-rule methods may deviate from correct values by as much as 2 percentage points. Therefore it is concluded that dependence should not be placed on results obtained by the chart or slide-rule methods for the acceptance or rejection of materials when the liquid limit as determined



A - LOCATION OF SPECIAL SCALE (BLOWS) WITH RESPECT TO B SCALE OF SLIDE RULE



B - SLIDE RULE SET FOR 21.4 PERCENT MOISTURE AT 20 BLOWS, INDICATING CALCULATED LIQUID LIMIT OF 20.8

Figure 4.—Slide rule with special scale for the calculation of liquid limit.

by either of these methods is within 2 percentage points of the specification limits. The data in figure 5 show that for the liquid limit range of 15 to 40, over 75 percent of the calculated values for both methods are within  $\pm 1$  percentage point and practically all are within  $\pm 2$  points of the values determined by the standard test procedure.

However, for the acceptance or rejection of soil-aggregate materials based on a specification having a maximum liquid limit of 25, a deviation of  $\pm 0.5$  percentage point from that value, or 2 percent of 25, is the maximum that can be permitted. In order to provide this accuracy, the one-point method needed further improvement. It was thought that this could be accomplished if the range of blows for groove closure was decreased.

**Accuracy of Methods Improved**

A statistical analysis was made of the test data to determine the range in number of blows for groove closure which would give results within 2 percent of those obtained by the AASHO standard method. The results of this analysis are shown in table 4 and a study of these data indicates that the desired level of accuracy can be accomplished by narrowing the range of the acceptable number of blows for groove closure from 17-36 to 22-28.

Three ranges in number of blows for groove closure, 29-35, 22-28, and 17-21 are indicated in table 4 to show the reproducibility obtained by chart and slide rule methods for six ranges in liquid limit. The number of tests studied in each of these ranges is also shown. To facilitate comparison of the accuracy of the

Table 2.—Frequencies of different size deviations of liquid limits derived by chart and slide-rule methods from those obtained by the AASHO standard method

Range of liquid limit	Number of one-point tests	Deviation <sup>1</sup> from AASHO standard	Frequency of deviations of—	
			Chart method	Slide-rule method
			Percent	Percent
15-25	171	0	76.6	84.8
		1	23.4	15.2
		2	0	0
26-40	209	0	76.1	77.0
		1	24.4	23.0
		2	.5	0
41-55	129	0	69.0	70.6
		1	27.1	27.9
		2	3.9	1.6
56-70	82	0	58.6	58.6
		1	40.2	40.2
		2	1.2	1.2
71-85	94	0	44.7	45.7
		1	47.9	48.9
		2	7.4	5.3
86-100+	74	0	39.2	35.2
		1	37.8	43.2
		2	20.2	18.9
		3	2.7	2.7
		4	0	0

<sup>1</sup> Deviations from the standard values, originally recorded to the nearest tenth of a percentage point, are here assigned to the nearest whole number.

**Table 3.—Values of the denominator of the curve formula corresponding to the number of blows required for groove closure in the liquid limit test**

Number of blows	Denominator of formula	Number of blows	Denominator of formula
15	1.066	28	0.985
16	1.059	29	.980
17	1.050	30	.976
18	1.043		
19	1.036	31	.972
20	1.029	32	.968
		33	.964
21	1.023	34	.960
22	1.017	35	.956
23	1.011		
24	1.005	36	.952
25	1.000	37	.948
		38	.945
26	.995	39	.942
27	.990	40	.939

methods for each of the three ranges in blows for groove closure, the number of cases having less than the indicated deviations is expressed on a percentage basis for each of the six ranges in liquid limit.

Within each of the ranges of liquid limit, the number of cases having less than each consecutive larger deviation is expressed on a cumulative percentage basis. For example, in the first line of table 4, under the chart method, for the 29-35 blow range and with a liquid limit range of 15-25, the percentages of test results deviating less than 0.3, 0.5, 0.8, 1.1, etc., percentage points from the true values are 42, 74, 90, 100, etc., respectively. The boldface values 42 and 74 are the percentages of test results deviating less than 0.3 and 0.5 percentage points, and are therefore within 2 percent of the maximum and minimum values of liquid limit range of 15-25. Similarly, other comparative values for any designated range in number of blows or liquid limits can be determined for either the chart or slide-rule methods.

A study of these comparative data in table 4 clearly shows that the greatest degree of accuracy for both methods is obtained in the 22-28 blow range and the least accuracy occurs in the 17-21 blow range. Within the

**Table 4.—Comparison of accuracy of chart and slide-rule methods<sup>1</sup>**

Range of blows for tests	Liquid limit ranges	Number of tests	Frequency of deviations from standard test values (in cumulative percentages)													
			Chart method deviations <sup>2</sup> (±) of							Slide-rule method deviations <sup>2</sup> (±) of						
			0.3-	0.5-	0.8-	1.1-	1.4-	1.7-	2.0+	0.3-	0.5-	0.8-	1.1-	1.4-	1.7-	2.0+
29-35	15-25	31	<b>42</b>	74	90	100	100	100	100	100	<b>81</b>	97	100	100	100	100
	26-40	37	51	84	<b>95</b>	100	100	100	100	100	65	<b>87</b>	100	100	100	100
	41-55	37	38	54	<b>92</b>	97	100	100	100	100	43	73	<b>95</b>	100	100	100
	56-70	22	32	41	86	<b>96</b>	<b>96</b>	100	100	100	23	50	86	<b>100</b>	<b>100</b>	100
	71-85	31	23	29	61	81	<b>90</b>	97	100	23	36	74	87	97	97	100
	86-100+	20	15	25	30	40	65	<b>95</b>	<b>100</b>	20	25	35	50	70	85	<b>100</b>
22-28	15-25	34	<b>88</b>	<b>100</b>	100	100	100	100	100	100	<b>91</b>	<b>100</b>	100	100	100	100
	26-40	27	85	<b>100</b>	<b>100</b>	100	100	100	100	100	93	<b>100</b>	<b>100</b>	100	100	100
	41-55	23	65	91	<b>100</b>	100	100	100	100	65	96	<b>100</b>	<b>100</b>	100	100	100
	56-70	13	62	92	100	<b>100</b>	<b>100</b>	100	100	69	82	100	<b>100</b>	<b>100</b>	100	100
	71-85	28	68	82	96	96	<b>100</b>	100	100	82	96	100	100	<b>100</b>	<b>100</b>	100
	86-100+	20	30	45	85	85	95	<b>100</b>	<b>100</b>	35	45	70	85	95	<b>100</b>	<b>100</b>
17-21	15-25	37	<b>51</b>	<b>81</b>	97	97	100	100	100	73	<b>95</b>	97	100	100	100	100
	26-40	43	56	77	<b>100</b>	100	100	100	100	58	84	98	100	100	100	100
	41-55	25	52	80	<b>88</b>	<b>96</b>	100	100	100	40	68	84	88	96	100	100
	56-70	22	36	50	73	<b>91</b>	<b>100</b>	100	100	36	50	68	82	<b>91</b>	100	100
	71-85	29	38	41	66	86	93	93	100	38	59	69	90	93	93	100
	86-100+	22	9	23	41	50	64	73	<b>100</b>	9	18	36	46	55	59	<b>100</b>

<sup>1</sup> Pairs of boldface values are the percentages of tests with deviations from standard test values less than 2 percent of the minimum and maximum liquid limit ranges shown.  
<sup>2</sup> Deviations in percentage points.

22-28 blow range there are no test values (see boldface results in table 4) calculated by either the chart or the slide-rule methods which exceed the maximum deviations based upon 2-percent limit of error in the liquid limit values determined by the AASHO standard method. The only exception was the deviation of less than 0.3 (which is 2 percent of the minimum value 15 in the 15-25 liquid limit range) which shows 88 percent for the chart method and 91 percent for the slide-rule method. This is insignificant, however, since it is the usual practice to report the liquid limit value to the nearest whole number. For this reason, the 22-28 blow range was selected as the limit to be used to obtain test results meeting the tolerance specified.

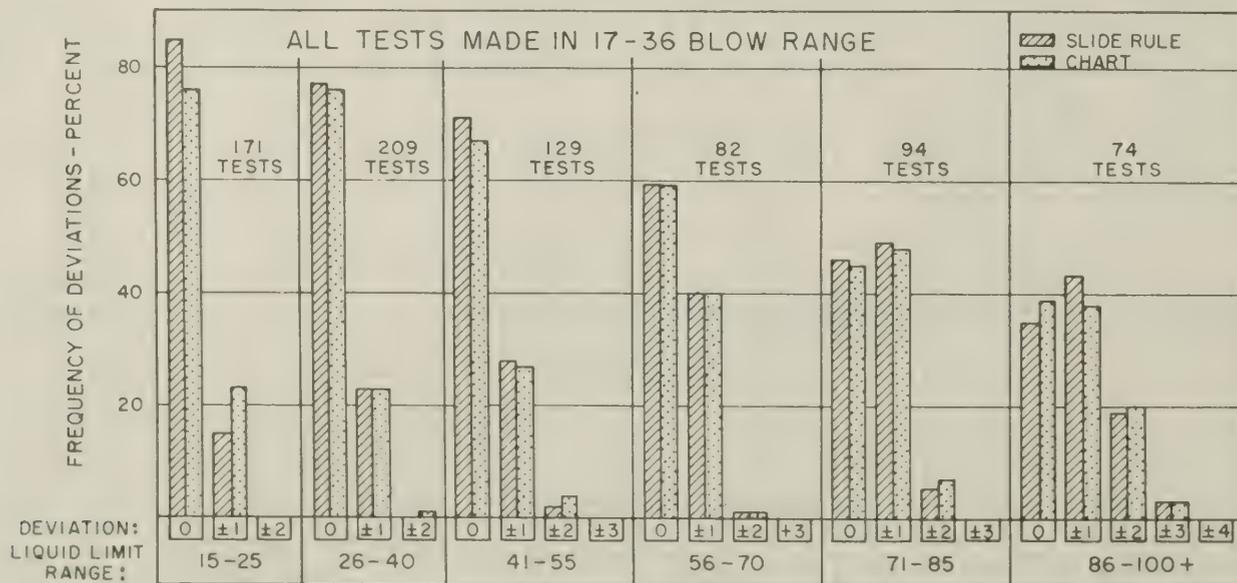
**Acceptability of Methods**

Thus, on the basis of the data obtained in this investigation, both the chart and slide-rule methods should be acceptable as alternatives for the standard methods provided (1) that procedure for the preparation of the soil

pat for test be that prescribed for the AASHO standard method, (2) that the acceptable number of blows for groove closure be within the 22-28 blow range, and (3) that at least two consecutive closures be observed before selecting the moisture sample for calculation of the liquid limit.

Under these conditions, the results of the slide-rule method have been consistently checked with those obtained by the AASHO standard method in the Bureau of Public Roads laboratory for more than 2 years. There appears to be no difficulty for technicians in the adjustment of the consistency within the 22-28 blow range.

It is suggested that other laboratories make similar comparative check studies, using local soils, to determine whether the accuracy of this improved one-point method of calculation can be established for a range of conditions even wider than those included in this study. If comparable accuracy is obtained by these proposed check tests, it would warrant the use of this method as a standard AASHO and ASTM method.



**Figure 5.—Percentage of chart and slide-rule tests deviating from the AASHO standard liquid limit.**

# A Study of Dispersing Agents for Particle-Size Analysis of Soils

BY THE PHYSICAL RESEARCH BRANCH  
BUREAU OF PUBLIC ROADS

Reported by ADOLPH M. WINTERMYER  
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*In the particle-size analysis of soils, mechanical agitation and the physico-chemical action of a dispersing agent are employed to disperse a soil sample in water and prepare a suspension for subsequent determination of particle size-groups by sedimentation techniques. The dispersing agent fulfills two functions vital to the accuracy of the analysis: it assists the mechanical treatment in the initial separation of naturally cemented or aggregated prime particles, and it provides conditions for normal sedimentation in the suspension during the test period by preventing flocculation (or other interfering effects) of the dispersed particles.*

*Since their introduction to analytical procedures a few decades ago, many different types of chemical dispersing agents have been employed, with varying results. At present, sodium metasilicate is widely used in soil testing for engineering purposes, and sodium hexametaphosphate, sodium hydroxide, and sodium carbonate are among those used for agricultural soils. However, the reported results of numerous studies do not afford a clear evaluation of effectiveness because of the lack of consistency in methods and techniques and, frequently, the narrow scope of experimental soils and dispersants. The investigation reported here was undertaken to provide this needed evaluation.*

*A large group of dispersing agents representing a wide range of chemical properties were tested with a broad selection of soils under closely controlled conditions of analysis. The resulting data showed sodium metasilicate to be relatively ineffectual in the dispersion of several soils and not suitable for general use. Several others, such as the carbonate, hydroxide, and oxalate of sodium, produced a high degree of dispersion with a few soils, but failed to stabilize the suspensions of others. Sodium polyphos and sodium tripolyphosphate were highly and about equally effective for all of the experimental soils representing many of the great soil groups of continental United States. Two others, sodium hexametaphosphate and sodium tetrakisphosphate, were only slightly less so. However, none of these four phosphates was effective with laterite, a soil of the tropics. Conversely, this soil was highly dispersed by trisodium phosphate and tetrasodium pyrophosphate, which were generally ineffective with members of the other soil groups.*

PARTICLE-SIZE or mechanical analysis is one of the oldest and most valuable test methods applied to soil materials. Its purpose is to determine the quantitative distribution of prime particles in several selected size-groups or separates, such as sand, silt, and clay. The resulting particle-size distribution data have long been used to characterize the physical makeup or texture of soils and have been especially important in soil classification for highway and other purposes.

Although details of analytical procedures vary considerably among different investigators, a required feature of all methods for particle-size analysis is the dispersion of the soil sample. A satisfactory dispersion requires complete separation of the prime particles of the soil or the reduction of agglomerates to sizes not larger than those of the smallest size-group to be determined, and

maintenance of the resulting soil suspension in a condition satisfactory for subsequent particle-size determinations. In methods now in general use, dispersion is accomplished by mechanical agitation of a soil-water mixture, in conjunction with an added chemical dispersing agent. Silt- and clay-size groups are determined from the resulting soil-water suspension by sedimentation techniques involving either hydrometer readings or pipetting and drying of aliquots. Sand groups are determined by standard sieving techniques.

The accuracy of the analysis is strongly affected by the degree to which the mechanical agitation and the dispersing agent satisfy the above two requirements. Mechanical dispersion is accomplished by stirring or shaking the soil-water mixture and, for this purpose, a high-speed motor-driven stirrer has been widely used in soil laboratories for many

years. A more recently developed air-jet device (12)<sup>1</sup> has been shown to produce an even more complete dispersion and also to minimize the attrition of prime particles which may occur with even a brief treatment with the motor-driven stirrer. The use of either device is now optional in AASHTO method T 88-49 (1).

## Purpose of Investigation

The investigation reported here was primarily concerned with the second requirement, or chemical phase, which has been far less well resolved. This concerns the physico-chemical activity of the dispersing agent in the soil-water mixture, which assists in the initial mechanical dispersion but is more particularly required for the maintenance of the resulting suspension. The problems involved in this part of the dispersion process result from inorganic and organic substances of clay and colloidal sizes. These adhere to and bind each other, as well as some of the larger particles, more or less firmly into aggregate structures which resist separation. In most highway soil materials, the clay and colloidal particles are largely inorganic in nature and consist chiefly of minute crystallites of one or more members of the clay mineral group. The crystal structures contain an excess of negative charges which attract positively charged ions such as H<sup>+</sup>, Ca<sup>++</sup>, and Na<sup>+</sup>. These, in turn, influence the tendency of the clay particles either to be dispersed or to remain in aggregates or floccules. The hydrogen and calcium forms of clays resist dispersion, whereas the particles of sodium clays are separated with relative ease.

Further, a dispersed suspension of soil particles in water may manifest colloidal properties such as gelation (syneresis) or coagulation and flocculation. Coagulation and flocculation hasten particle sedimentation, and gelation retards it. In the first case, hydrometer readings made in the soil suspension for particle-size measurements yield low results for the finer particles, and the analysis makes the soil appear coarser than it actually is; in the second, the reverse is true.

Most dispersing agents provide sodium ions to replace the flocculating cations from the

<sup>1</sup> Italic numbers in parentheses refer to the list of references on page 62.

soil clay, and anions such as hydroxyl, oxalate, carbonate, phosphate, or silicate to inactivate the replaced cations. Or, if instead of an electrolyte, a lyocatic (protective) colloid such as gum acacia is used, dispersion may be aided and the suspension maintained by the action of the colloid in forming protective layers around the clay particles. As often happens in practice, the dispersing agent may fail to provide these effects, with the result that particle sedimentation is interfered with and measurements of the silt and clay fractions are invalidated.

In the long history of particle-size analysis, many different chemical dispersants have been used with varying success and acceptance. Among the more common are ammonium hydroxide and the carbonate, oxalate, and hydroxide of sodium. Sodium hexametaphosphate is now widely used with agricultural soils (7, 11) and AASHO method T 88-49 (1) specifies sodium metasilicate. The extensive literature reveals the extreme variability under which analyses have been made, the variations including experimental procedures and apparatus, types and concentrations of electrolytes, types and amounts of soils, and pretreatments of samples for removal of organic matter, soluble salts, or exchangeable bases. Rarely are comparative data presented for more than one or two dispersing agents, and the soils studied are usually severely limited in number and variety. Thus, for the most part, the literature reveals an inadequate testing and evaluation of dispersing agents with regard to degree of dispersion obtained and to suitability for use with a wide variety of soils. It can only be concluded that, in general, the several dispersing agents used in the past vary considerably in both respects. Some are of severely limited value and none has been found suitable for use with all soils.

### Summary and Conclusions

This report presents the principal findings of a study designed to evaluate the effectiveness of a large group of dispersing agents with a wide variety of soils under closely controlled conditions of analysis. For purposes of comparison, data obtained by the use of AASHO method T 88-49 are also presented. The criterion adopted for the degree of dispersion obtained is the percentage value for the size-group of particles having a diameter of 1 micron (0.001 mm.) or less. This is determined from the 24-hour hydrometer reading and is termed the "dispersion value." This size-group was selected because it is the smallest now determined in particle-size analyses for highway purposes. Further, its upper-limit is well below the 2-micron value commonly accepted for the clay fraction.

Nineteen chemical dispersing agents were tested to determine their effectiveness in the dispersion of soils and the stabilization of soil suspensions for particle-size analysis. Dispersions were made in an air-jet dispersion cup and with a high-speed motor stirrer. In all, 14 soils and 2 clay minerals were tested. Both soils and dispersing agents were selected to include a wide range of properties and

conditions of the respective materials. In all tests, complete particle-size analyses were made and the usual size-groups were determined by the hydrometer method and with standard sieves. Except for variations in kind and amount of dispersant, details of the analyses conformed to the standard AASHO method T 88-49 (1). Sodium metasilicate, of the AASHO method, was among the dispersants tested. Principal findings and conclusions from the investigation are as follows:

Thirteen of the dispersing agents were found to be generally unsatisfactory. Several failed to stabilize the suspensions of one or more soils, others frequently produced low dispersion values, and some were ineffective in both respects.

Sodium metasilicate was among the less satisfactory dispersants. Although it failed to stabilize the suspension of only one soil, many of its dispersion values were seriously low. Its use as a general soil dispersing agent should be discouraged.

Widely effective dispersing agents were found only among the phosphate materials. Two of these, sodium polyphos and sodium tripolyphosphate, were highly effective with all soils except one, a laterite. Sodium hexametaphosphate and sodium tetraphosphate were only slightly less effective.

The highest dispersion values for a laterite soil were obtained with trisodium phosphate and tetrasodium pyrophosphate. For most other soils, however, the first was unsatisfactory and the second produced dispersion values that were only moderately high.

From the standpoint of special groupings, such as podzolic, illitic, kaolinitic, and montmorillonitic soils, the four phosphates noted in the second paragraph above were consistently highly effective, though their relative ratings varied somewhat among the groups. For general use, sodium polyphos is rated best and sodium tripolyphosphate is a close second. Their unsuitability for the dispersion of a true laterite soil does not extend to related partially laterized soils.

### Materials Studied

The 19 dispersing agents selected for this experimental study are listed in table 1 divided among seven subgroups according to chemical composition. These include most of the materials which have been widely used for soil dispersion. A few of the phosphates are new to soil testing, and others such as Lomar PW and Marasperse CB have not been employed with soils but are finding application as industrial dispersing agents.

With a few exceptions, the chemical reagent grade of each material was used. Sodium tripolyphosphate and sodium polyphos were of technical grade and gum acacia and gum ghatti were of pharmaceutical quality. Although the grade of Marasperse CB is not known, its manufacturer has stated that it is a highly purified product. All are readily available at a cost not too high to prohibit their use in routine laboratory work.

The soils chosen for analysis are listed and described in table 2. In addition to the soil names, when known, their liquid and plastic limits and classifications, as derived from the

Table 1.—Experimental dispersing agents

Identification	Dispersing agent		Stock solution		Type of reaction
	Name	Formula	Concentration	Grams per liter	
A	Sodium metasilicate (sodium silicate).	$\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$	0.4N	56.8	Supplies dispersive cation and alkaline reaction. Hydrolyzes to silicic acid which acts as protective colloid (4) and precipitates Ca and Mg.
B	Sodium hexametaphosphate <sup>1</sup>	$\text{Na}_6\text{P}_6\text{O}_{18}$ or $(\text{NaPO}_3)_6$	0.4N	40.8	Forms soluble undissociated complexes with many cations and prevents flocculation effects. Also supplies a dispersive cation.
C	Trisodium phosphate	$\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$	0.4N	50.7	
D	Tetrasodium pyrophosphate	$\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$	0.4N	44.6	
E	Sodium tripolyphosphate <sup>2</sup>	$\text{Na}_3\text{P}_3\text{O}_{10}$	0.4N	29.4	
F	Sodium tetraphosphate <sup>3</sup>	$\text{Na}_4\text{P}_4\text{O}_{13}$	0.4N	31.3	
G	Sodium polyphos <sup>2</sup>	$\text{Na}_{12}\text{P}_{10}\text{O}_{31}$	0.4N	36.1	
H	Victamide	(4)	0.4N	38.6	
I	Sodium oxalate	$\text{Na}_2\text{C}_2\text{O}_4$	0.4N	26.8	
J	Lithium oxalate	$\text{Li}_2\text{C}_2\text{O}_4$	0.4N	20.4	
K	Ammonium oxalate	$(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$	0.4N	28.4	
L	Sodium hydroxide	$\text{NaOH}$	0.4N	16.0	Supplies dispersive cation and alkaline reaction.
M	Lithium hydroxide	$\text{LiOH}$	0.4N	9.6	
N	Sodium carbonate	$\text{Na}_2\text{CO}_3$	0.4N	21.2	Supplies dispersive cation and alkaline reaction.
O	Lithium carbonate	$\text{Li}_2\text{CO}_3$	0.1N	3.7	
P	Gum acacia	Complex colloidal polysaccharide.	0.13%	1.3	Prevents coalescence of particles by providing protective hydration layer.
Q	Gum ghatti	do	0.13%	1.3	
R	Lomar PW	Complex sulfonate <sup>5</sup>	0.4N	95.3	Produces repelling negative charges on suspended particles.
S	Marasperse CB	Complex sulfonate <sup>6</sup>	0.33%	3.3	

<sup>1</sup> Calgon, buffered with  $\text{Na}_2\text{CO}_3$ .  
<sup>2</sup> Sodium tripolyphosphate and sodium polyphos are products of the fusion of various combinations of phosphates, purchased from Blockson Chemical Co., Joliet, Ill.  
<sup>3</sup> Sodium tetraphosphate is sold under the name of Quadrafos by Rumford Chemical Works, Rumford, N. J.  
<sup>4</sup> Victamide is an ammonium amido-polyphosphate furnished by the Victor Chemical Works, Chicago, Ill. Its exact formula has not been released but  $(\text{NH}_4)_2\text{P}_2\text{O}_4 \cdot \text{NH}_4\text{NHP}_2\text{O}_5(\text{NH}_4)_2$  was used to calculate the weight needed for a 0.4N stock solution.  
<sup>5</sup> Lomar PW is the sodium salt of a condensed mono-naphthalene sulfonic acid, furnished by Jaques & Co., Passaic, N. J. The formula  $(\text{C}_{10}\text{H}_7\text{SO}_3\text{NaCH}_2)_2 \cdot \text{C}_{10}\text{H}_7\text{SO}_3\text{Na}$  was used to calculate the weight needed for a 0.4N stock solution.  
<sup>6</sup> Marasperse CB is an indefinite mixture of partially desulfonated sodium lignosulfonates furnished by the Maratho Corp., Rothschild, Wis.

Table 2.—Experimental soils

Soil No.	Series name	Soil group	Geographical source	Liquid limit	Plasticity index	pH (1-1 mix)	HRB classification and group index
1	Iredell	Yellow podzolic	Fairfax Co., Va.	88	62	5.7	A-7-6(20)
2	Cecil	Red podzolic	Lee Co., Ala.	63	22	4.7	A-7-5(16)
3	Tenn. I	Red podzolic	Sevier Co., Tenn.	37	8	5.1	A-4(6)
4	Tenn. II	Yellow podzolic	Sevier Co., Tenn.	38	9	4.9	A-4(6)
5	Sharkey	Alluvium from podzolic materials	Knox Co., Ind.	47	24	6.7	A-7-6(15)
6	Nipe	Laterite	Mayaguez, P. R.	44	13	4.9	A-7-5(10)
7	Miami	Gray-brown podzolic	Tippacanoe Co., Ind.	33	18	7.6	A-6(10)
8	Washo	Lake sediment in arid climate	Oneida Co., Idaho	35	9	7.8	A-4(8)
9	Houston	Rendzina (blackland)	Delta Co., Tex.	54	32	7.0	A-7-6(19)
10	Yazoo	Alluvium of lower Miss. Valley	Hinds Co., Miss.	91	65	7.8	A-7-6(20)
11	Wabash	Alluvium of Miss. Delta	Madison Parish, La.	68	40	6.7	A-7-6(20)
12	Salt Flat I	Alkali (high Na <sub>2</sub> SO <sub>4</sub> )	Lyon Co., Nev.	58	35	8.6	A-7-6(20)
13	Salt Flat II	Alkali (high NaCl)	Churchill Co., Nev.	23	8	9.4	A-4(8)
14	Putnam	Planosol (clay layer accumulation)	Missouri	50	30	6.5	A-7-6(18)
15	Bentonite	Clay mineral montmorillonite	Wyoming	347	303	8.6	A-7-5(20)
16	Kaolin	Clay mineral kaolinite	Georgia	49	15	8.7	A-7-5(12)

<sup>1</sup> Series name not known; soil occurs in unmapped area.

limit values and the percentages passing the No. 40 and No. 200 sieves, are given as further descriptive information. All of the samples are from the B or C horizons, since this is the part of the soil profile usually used in highway construction.

The soils selected represent a fairly wide range of genetical conditions and include members of many of the great soil groups of the world. Only one of the samples, Nipe (No. 6), is from outside the United States. This is a true laterite, a soil group common to the tropics but not found in this country. Two clay mineral samples, a bentonite and a kaolin (Nos. 15 and 16), were included for testing since they are relatively pure examples of two of the three chief clay mineral groups common to soils. An illite sample was not available in sufficient quantity for the testing of this clay mineral group as such, but illite occurs as a major clay mineral constituent in some of the soils included in the list.

Samples were dispersed (at an air pressure of 20 p.s.i. from a laboratory compressed-air line) with a Model A air-jet dispersion cup which has been described in a previous report (12) and approved for use in AASHTO method T 88-49 (1).

Densities of the soil suspensions were measured with an early model of the streamlined Bouyoucos hydrometer, calibrated for use at 67° F. Density readings were corrected for deviations from this temperature and for the presence of the dispersing agent.

**Procedure and Methods**

Essentially, the experimental work consisted of the following tests:

1. Soils 1-6 were dispersed in the air-jet dispersion cup with each of 19 dispersants, which were added in 5- or 10-ml. increments of stock solution to replicate portions of each soil.

2. Soils 7-14 and the clay minerals (15 and 16) were dispersed in the air-jet dispersion cup with a single concentration of each of the phosphates.

3. Soils 1-14 were dispersed with the motor stirrer and sodium metasilicate solution.

For those tests made by the use of the air-jet dispersion cup, a 50-g. portion of air-dry soil passing the 2-mm. (No. 10) sieve was soaked overnight in a beaker containing about

75 ml. of distilled water. The mixture was then transferred to the dispersion cup with the use of an additional 75 ml. of water from a wash bottle; the predetermined volume of dispersant stock solution was added; and, with the air-pressure adjusted to 20 p.s.i., the mixture was dispersed for 5 minutes with soils having a plasticity index of 5 and less, 10 minutes with those between 6 and 20, and 15 minutes with those greater than 20. These dispersion times were established previously (12) from tests of a large number of soils. The dispersed mixture was then transferred to a 1,000-ml. volumetric cylinder and made up to volume.

In accord with the details of the AASHTO method, hydrometer readings were made in the suspension at 2-, 60-, and 1,440-minute intervals for the silt, clay, and "colloid" size-groups and the mixture was then passed through standard sieves for separation of the sand groups. Sediment volumes were recorded at the time of the hydrometer readings and the suspensions were examined visually for evidence of flocculation or other interfering effects. Measurements of pH were obtained for each soil (1-1 soil-water mixture), dispersant solution, and soil-water-dispersant mixture, using a glass electrode and a Beckman Model G pH meter.

For the tests made with the motor stirrer, the mixture was given a 1-minute dispersion

in the prescribed cup of this device with 20 ml. of a 3° Baumé (approximately 0.2N) sodium metasilicate solution. Otherwise, the details of the analysis were the same as those given above.

Stock solutions of those dispersing agents having a definite chemical formula were 0.4N, except that of lithium carbonate, which was limited to 0.1N by low solubility. Those of the gums were 0.13 percent and that of Marasperse CB was 0.33 percent, by weight. Weights used for a liter of stock solution are shown in table 1. The volumes used in individual tests ranged from 5 ml. to a maximum at which no further increase in dispersion was measured.

**Evaluation of Dispersants**

Although percentage values of the usual size-groups were determined in all of the tests, the present report is chiefly concerned with those of the less-than-0.001-mm. size-group, the dispersion values. In the majority of cases in the first series of tests, the dispersion values increased with increasing concentrations of the dispersant, and reached a maximum. With further increases in concentration, varying for different soils and dispersants, the degree of dispersion either persisted at the maximum value or decreased to lower values. Several examples of these effects are illustrated by the curves in figure 1. The concentrations at which maximum values occurred are referred to as the "optimum concentration range." Those yielding values within 1 percentage point of the maximum value are termed the "practical concentration range." Several other effects which also occurred in this series of tests were flocculation at low and high but not at medium concentrations, gelling at certain concentrations, and failure of suspensions over the entire concentration range.

The dispersion values obtained for soils 1-6 at optimum concentrations of each of the dispersants are given in table 3. The figure in parentheses adjoining each dispersion value represents the deviation of that value from the maximum of the 20 dispersion values obtained for that soil. The symbol *F* indicates failure of the suspension due to flocculation or other

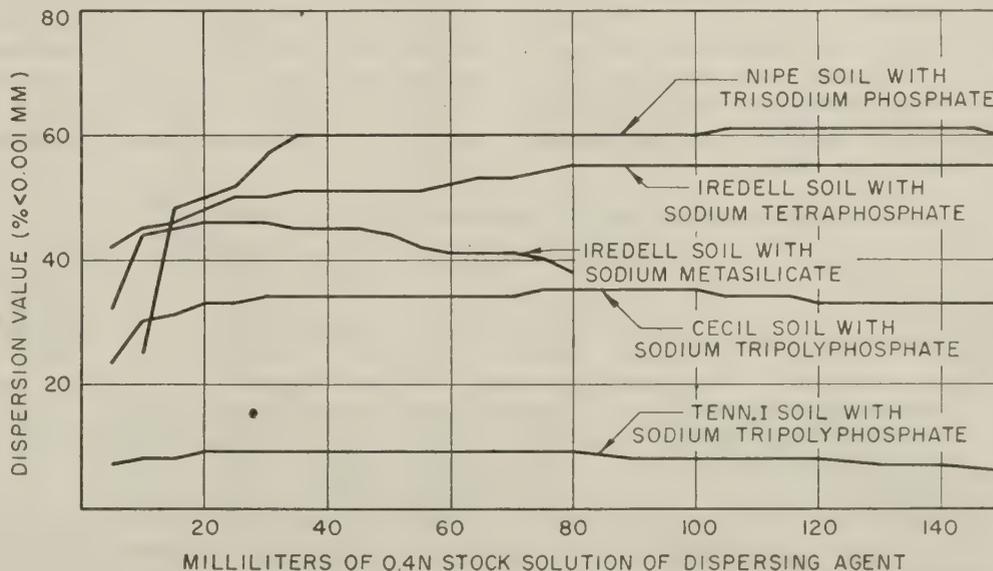


Figure 1.—Effect of dispersing agent concentration on dispersion value.

**Table 3.—Effectiveness of 19 dispersing agents with soils Nos. 1-6**

Dispersing agent		Dispersion values <sup>1</sup> (percentage of the less-than-0.001-mm. group) obtained with the air-jet dispersion cup at the optimum concentrations of the dispersing agents for soil—						Average deviation from maximum dispersion value (percentage points) for Podzolic soils (Nos. 1-5)
Identification	Name	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	
A	Sodium metasilicate.....	46 (11)	33 (3)	7 (3)	6 (2)	28 (8)	51 (10)	5.4
B	Sodium hexametaphosphate.....	55 (2)	36 (0)	10 (0)	7 (1)	34 (2)	52 (9)	1.0
C	Trisodium phosphate.....	52 (5)	F	F	F	30 (6)	61 (0)	F
D	Tetrasodium pyrophosphate.....	53 (4)	34 (2)	8 (2)	5 (3)	33 (3)	60 (1)	2.8
E	Sodium tripolyphosphate.....	54 (3)	35 (1)	9 (1)	7 (1)	35 (1)	56 (5)	1.4
F	Sodium tetraphosphate.....	55 (2)	36 (0)	10 (0)	8 (0)	33 (3)	52 (9)	1.0
G	Sodium polyphos.....	56 (1)	33 (3)	8 (2)	6 (2)	34 (2)	48 (13)	2.0
H	Victamide.....	54 (3)	34 (2)	ND	ND	ND	42 (19)	2.5
I	Sodium oxalate.....	53 (4)	F	F	5 (3)	32 (4)	F	F
J	Lithium oxalate.....	52 (5)	F	F	3 (5)	34 (2)	F	F
K	Ammonium oxalate.....	49 (8)	F	F	4 (4)	34 (2)	F	F
L	Sodium hydroxide.....	54 (3)	F	F	4 (4)	34 (2)	F	F
M	Lithium hydroxide.....	53 (4)	34 (2)	F	3 (5)	36 (0)	59 (2)	F
N	Sodium carbonate.....	50 (7)	F	F	3 (5)	33 (3)	57 (4)	F
O	Lithium carbonate.....	57 (0)	F	F	3 (5)	33 (3)	41 (20)	F
P	Gum acacia.....	F	16 (20)	5 (5)	4 (4)	21 (15)	F	F
Q	Gum ghatti.....	44 (13)	15 (21)	9 (1)	5 (3)	23 (13)	14 (47)	10.2
R	Lomar PW.....	F	F	9 (1)	5 (3)	F	44 (17)	F
S	Marasperse CB.....	52 (5)	22 (14)	9 (1)	7 (1)	28 (8)	43 (18)	5.8
AASHO method with sodium metasilicate and 1 min. in motor stirrer.		29 (31)	22 (14)	5 (5)	5 (3)	13 (23)	20 (41)	15.2

<sup>1</sup> Numbers in parentheses are deviations (in percentage points) of the given dispersion value from the maximum value obtained for that soil.

F=suspension failure due to flocculation or other colloidal effect; ND=value not determined.

colloidal effects at all concentrations of the dispersant.

As indicated by the *F* symbols, ten of the dispersants failed at all concentrations with one or more soils. Two others, gum ghatti and Marasperse CB, maintained the suspensions of all six soils, but were highly ineffectual, as shown by their deviation values. For at least one soil, however, each of these twelve dispersants produced dispersion values equal to or reasonably near the maximum value. This explains why, despite their failure with certain soils, several of these have been considered satisfactory in laboratories where the soils tested are closely related or of limited variety.

For each soil, wide variations occur among the dispersion values given by the several dispersing agents. This indicates that close agreement ( $\pm 1$  percentage point) between the values from duplicate tests with the same soil does not insure that a specific dispersing agent has provided a high degree of dispersion since other duplicate samples of the same soil, dispersed with a different agent, will also yield closely agreeing dispersion values, though they are higher or lower in degree.

With the podzolic soils 1-5, the majority of the phosphates provide a combination of high dispersion values (low deviations) and freedom of suspensions from flocculation or other colloidal effects. Of the phosphates, B, E, F, and G were highly effective and D and H were only slightly less so. However, C failed at all concentrations with three of the five soils.

With the laterite soil (No. 6), a high degree of dispersion was obtained from only five of the dispersants, among which were three phosphates: C, D, and E. Trisodium phosphate (C), which was ineffectual with the podzolic soils, gave the highest dispersion value obtained. This indicates that the dispersion of laterites presents a different problem from that of the podzolic soils.

For most soils, sodium metasilicate yielded low dispersion values, at optimum concentrations in the air-jet dispersion cup (line 1, table 3), and at the single concentration with the motor stirrer (bottom line of table 3). It has, however, the advantage of preventing flocculation in tests of all six soils. The dispersion values for soils 2 and 5 also provide data for a valid comparison of the effectiveness of the air-jet dispersion cup and the motor

**Table 4.—Practical concentrations<sup>1</sup> of the phosphates**

Dispersing agent		Practical concentration range for soil—					Practical concentration for podzolic soils 1-5
Identification	Name	No. 1	No. 2	No. 3	No. 4	No. 5	
B	Sodium hexametaphosphate.....	85-150	120-150	105-150	25-150	135-150	140
C	Trisodium phosphate.....	40-55	F	F	F	20-100	55
D	Tetrasodium pyrophosphate.....	90-140	5-110	20-100	25-140	60-150	100
E	Sodium tripolyphosphate.....	25-100	30-115	10-120	15-100	45-150	80
F	Sodium tetraphosphate.....	75-150	50-150	55-150	75-150	65-150	140
G	Sodium polyphos.....	10-115	30-150	10-80	10-85	30-150	75
H	Victamide.....	20-95	10-140	ND	ND	ND	90

<sup>1</sup> Expressed as ml. of stock solution.

F=suspension failure due to flocculation or other colloidal effect; ND=value not determined.

stirrer for mechanical dispersion since the single concentration of sodium metasilicate employed in the AASHO method is within the optimum concentration range for both soils in the air-jet dispersion cup. Dispersion values from the air-jet dispersion cup are higher by 11 percentage points with soil 2, and 15 points with soil 5. For the other soils, values from the air-jet dispersion cup range from 1 to 31 percentage points higher, but concentrations of the dispersant are not equivalent.

### Further Studies of Phosphates

In the second series of tests, soils 7-14 and the clay minerals bentonite and kaolin (15 and 16) were dispersed at the "practical concentration" of each of the several phosphates. This concentration, expressed as a volume of stock solution, was determined individually for each phosphate from the volume ranges in tests of soils 1-5 that gave dispersion values within 1 percentage point of the maximum. Practical concentration values and the practical volume ranges from which they were derived are listed in table 4.

The results of the second series of tests, shown in table 5, agree in general with those pertaining to the phosphates in the first series and reveal several points of further information:

Sodium tripolyphosphate and sodium polyphos (E and G) are highly effective for the soils and clay minerals, having six optimum dispersion values (zero deviations) out of a possible ten, maximum deviations of 5 and 3 percentage points, and low average deviations of 1.2 and 1.0.

Sodium hexametaphosphate and sodium tetraphosphate (B and F) are somewhat less effective, as shown by their average deviation values of 2.9 and 2.6. This is undoubtedly related to their higher deviations with the clay minerals: 21 and 10 for bentonite, and 4 in both cases for kaolin. Approximately the same degree of effectiveness is shown by tetrasodium pyrophosphate (D) for most of the soils, but two deviations of 8 points raise its average to 4.4 which approximates its dispersion results with the clay minerals.

Trisodium phosphate and Victamide (C and H) each failed to maintain the suspensions of three soils and was only moderately effective with the others. Victamide, however, produced the optimum dispersion value for kaolin but was ineffective with bentonite, and trisodium phosphate was ineffective with both of the clay minerals.

Sodium metasilicate (A) was even less effective than in the previous series of tests, with a suspension failure with one soil and very high deviation values, varying from 10 to 34, with the others.

Summarizing the results obtained with the clay minerals, bentonite was highly dispersed by only sodium tripolyphosphate and sodium polyphos (E and G). Kaolin was highly dispersed by both of these and Victamide (H), and moderately well by sodium hexametaphosphate, tetrasodium pyrophosphate, and sodium tetraphosphate (B, D, and F). It is significant that only sodium tripolyphosphate

**Table 5.—Effectiveness of phosphates with soils Nos. 7-14 and the clay minerals bentonite and kaolinite (Nos. 15 and 16)**

Dispersing agent			Dispersion value <sup>1</sup> (percentage of less-than-0.001-mm group) for soil or clay mineral—										Deviation from maximum dispersion value (average for soils 7-14)	
Identification	Name	Concentration (ml. of stock solution)	No. 7	No. 8	No. 9	No. 10	No. 11	No. 12	No. 13	No. 14	No. 15	No. 16		
B	Sodium hexametaphosphate.....	140	25 (3)	20 (3)	40 (3)	57 (3)	40 (5)	44 (4)	28 (2)	38 (0)	63 (21)	35 (4)	2.9	
C	Trisodium phosphate.....	55	25 (3)	18 (5)	39 (4)	F	39 (6)	F	F	38 (0)	F	32 (7)		
D	Tetrasodium pyrophosphate.....	100	26 (2)	15 (8)	39 (4)	56 (4)	41 (4)	40 (8)	27 (3)	36 (2)	78 (6)	35 (4)	4.4	
E	Sodium tripolyphosphate.....	80	28 (0)	23 (0)	43 (0)	58 (2)	42 (3)	43 (5)	30 (0)	38 (0)	82 (2)	39 (0)		
F	Sodium tetraphosphate.....	140	25 (3)	20 (3)	41 (2)	57 (3)	43 (2)	45 (3)	27 (3)	36 (2)	74 (10)	35 (4)	1.2	
G	Sodium polyphos.....	75	26 (2)	20 (3)	41 (2)	60 (0)	45 (0)	48 (0)	29 (1)	38 (0)	84 (0)	39 (0)		
H	Victamide.....	90	22 (6)	20 (3)	30 (13)	F	F	F	28 (2)	37 (1)	68 (16)	39 (0)	1.0	
AASHO method with sodium metasilicate and 1 min. in motor stirrer.....			20	8 (20)	7 (16)	9 (34)	34 (26)	14 (31)	F	20 (10)	10 (28)	ND	ND	23.6

<sup>1</sup> All values in parentheses are deviations (in percentage points) of the given dispersion value from the maximum value obtained for that soil. F=suspension failure due to flocculation or other colloidal effect; ND=value not determined.

and sodium polyphos (E and G) were highly effective for both of the clay minerals.

### The Five Effective Phosphates

In order to compare the essential results obtained by using the most effective phosphates with all of the experimental soils and with certain special groupings of soils, some of the data were selected from tables 3 and 5 and rearranged and summarized in table 6. Due to their failure with several suspensions, phosphates C and H were not included.

Comparison of the results of tests on all of the soils, 1-14, shows sodium tripolyphosphate (E) to be most effective, with an average deviation value of 1.4 percentage points, followed closely by sodium polyphos, sodium tetraphosphate, sodium hexametaphosphate, and tetrasodium pyrophosphate (G, F, B, and D) in the order of their average deviation values. The maximum deviation values show that each dispersant gives a high deviation with at least one of the soils of the group.

For the soils in the groups 1-5 and 7-14, from which the laterite is excluded, the best dispersant is sodium polyphos (G), which has a very low average deviation value of 0.6. Sodium tripolyphosphate, sodium tetraphosphate, and sodium hexametaphosphate (E, F, and B) follow in the order given. The average deviation of sodium pyrophosphate (D) is 3.6 percentage points, which is significantly higher than that of the others.

For the podzolic soils, sodium tripolyphosphate, sodium hexametaphosphate, and sodium tetraphosphate (E, B, and F) are almost equal in effectiveness, with very closely agreeing average deviation values of 1.2, 1.3,

and 1.3. Those of sodium polyphos (G) are slightly higher but probably not significantly so. Although tetrasodium pyrophosphate (D) is again the least effective, it is noteworthy that in this group of soils none of the phosphates produced an inordinately high maximum deviation value.

As previously noted, an illite clay mineral, per se, was not available in sufficient quantity for testing. However, data for soils 5, 7, 10, and 11, which are reported as highly illitic (2, 5), are combined in table 6 for an examination of dispersion results where illite is the principal (in percentage) clay mineral. These soils may also be considered as a group containing mixtures of the three chief clay minerals, since the reported analyses (referred to above) indicate that in addition to illite, each contains about 10 percent of kaolinite and 25 percent of montmorillonite. Sodium polyphos (G) again gave highest dispersion, with an average deviation of 1.0; sodium tripolyphosphate (E) is next, with 1.8 percentage points; and sodium tetraphosphate, sodium hexametaphosphate, and tetrasodium pyrophosphate (F, B, and D) follow as a less effective group. Here, again, maximum deviations are not high.

Since only two examples of kaolinitic soils, Cecil and Nipe (2 and 6), are included in the experimental list and since these also contain considerable amounts of iron oxides (3, 6), the specific influence of kaolinite in soils is not precisely defined by the data. However, deviation values given in table 6 for Cecil soil and kaolin (2 and 16), indicate that the five phosphates listed are relatively highly and about equally effective in dispersing these kaolinitic materials. Generally higher devia-

tions for the laterite (Nipe) may be due to the very high percentage of iron oxides present in this soil. However, the moderately high iron oxide content of Cecil soil has not similarly influenced its dispersion characteristics.

Results of the dispersion of bentonite show little resemblance to those of Iredell, Houston, and Putnam soils (1, 9, and 14) in table 6 which have been reported (6, 10) to be highly montmorillonitic. These three soils are highly dispersed by each of the phosphates, though only sodium tripolyphosphate and sodium polyphos (E and G) are highly effective with bentonite. Tetrasodium pyrophosphate (D) is moderately effective, and sodium hexametaphosphate and sodium tetraphosphate (B and F) are unsatisfactory. All of the phosphates were very highly effective with Putnam (14), a clay-pan soil with a clay fraction consisting largely of beidellite, a species of montmorillonite (8).

### Other Observations

Several examples of the specific size-groups affected during the later stages of the dispersion process are given in tables 7 and 8. Those in table 7 are of soils dispersed in the air-jet dispersion cup with three dispersant concentrations, yielding low, medium, and high degrees of dispersion. The size-groups sustaining losses when the dispersion is increased are found to vary from one soil to another. Losses in Cecil and Iredell occur chiefly in the 0.05-0.005-mm. and 0.005-0.001-mm. groups, and Nipe sustains losses in all of the groups above the less-than-0.001-mm. size. Although a certain amount of attrition of prime particles undoubtedly occurs with any soil,

**Table 6.—Effectiveness of phosphates with all soils tested and with certain groups of soils**

Dispersing agent		Deviation of the dispersion values from the maximum (percentage points)																				
		All soils (Nos. 1-14)			All soils except laterite (Nos. 1-5 and 7-14)			Podzolic soils (Nos. 1-5 and 7)			Illitic soils (Nos. 5, 7, 10, 11)			Kaolinitic soils			Kaolinite (No. 16)	Montmorillonitic soils			Montmorillonite (Bentonite) (No. 15)	
Identification	Name	Max.	Min.	Avg.	Max.	Min.	Avg.	Max.	Min.	Avg.	Max.	Min.	Avg.	No. 2	No. 6	Avg.		No. 1	No. 9	No. 14		Avg.
B	Sodium hexametaphosphate.....	9	0	2.5	5	0	2.0	3	0	1.3	5	2	3.2	0	9	4.5	4	2	3	0	1.7	21
D	Tetrasodium pyrophosphate.....	8	1	3.4	8	2	3.6	4	2	2.7	4	2	3.2	2	1	1.5	4	4	4	2	3.3	6
E	Sodium tripolyphosphate.....	5	0	1.4	5	0	1.2	3	0	1.2	3	1	1.8	1	5	3.0	0	3	0	0	1.0	2
F	Sodium tetraphosphate.....	9	0	2.4	3	0	1.8	3	0	1.3	3	2	2.5	0	9	4.5	4	2	2	2	2.0	10
G	Sodium polyphos.....	13	0	2.1	3	0	.6	3	1	2.0	2	0	1.0	3	13	8.0	0	1	2	0	1.0	0

**Table 7.—Effect of low, medium, and high degrees of dispersion (three levels of dispersant concentration) on the amount<sup>1</sup> of specific size-groups**

Size group (mm.)	Amount found when dispersant concentration is—			Differences			Amount found when dispersant concentration is—			Differences		
	Low (a)	Medium (b)	High (c)	b-a	c-b	c-a	Low (a)	Medium (b)	High (c)	b-a	c-b	c-a
CECIL SOIL (No. 2)												
	Dispersed with sodium tripolyphosphate (a=10 ml.; b=20 ml.; c=60 ml.)						Dispersed with sodium polyphos (a=10 ml.; b=30 ml.; c=100 ml.)					
2-0.42.....	4	5	4	+1	-1	0	4	4	4	0	0	0
0.42-0.25.....	8	7	8	-1	+1	0	8	8	8	0	0	0
0.25-0.074.....	20	20	19	0	-1	-1	19	19	19	0	0	0
0.074-0.05.....	4	4	3	0	-1	-1	5	5	5	0	0	0
0.05-0.005.....	25	21	22	-4	+1	-3	24	23	21	-1	-2	-3
0.005-0.001.....	16	13	9	-3	-4	-7	16	11	10	-5	-1	-6
Less than 0.001.....	23	30	35	+7	+5	+12	24	30	33	+6	+3	+9
NIPE SOIL (LATERITE, No. 6)												
	Dispersed with trisodium phosphate (a=50 ml.; b=60 ml.; c=120 ml.)						Dispersed with sodium polyphos (a=10 ml.; b=20 ml.; c=60 ml.)					
2-0.42.....	6	5	3	-1	-2	-3	13	10	8	-3	-2	-5
0.42-0.25.....	2	1	1	-1	0	-1	5	5	3	0	-2	-2
0.25-0.074.....	5	4	3	-1	-1	-2	10	9	6	-1	-3	-4
0.074-0.05.....	1	1	0	0	-1	-1	4	3	2	-1	-1	-2
0.05-0.005.....	18	15	15	-3	0	-3	24	21	20	-3	-1	-4
0.005-0.001.....	16	17	17	+1	0	+1	10	12	13	+2	+1	+3
Less than 0.001.....	52	57	61	+5	+4	+9	36	40	48	+4	+8	+12
IREDELL SOIL (No. 1)												
	Dispersed with sodium hexametaphosphate (a=10 ml.; b=40 ml.; c=250 ml.)						Dispersed with tetrasodium pyrophos (a=10 ml.; b=30 ml.; c=240 ml.)					
2-0.42.....	10	9	9	-1	+1	0	10	10	10	0	0	0
0.42-0.25.....	2	2	2	0	0	0	2	2	2	0	0	0
0.25-0.074.....	8	9	9	+1	-1	0	8	8	8	0	0	0
0.074-0.05.....	2	2	1	0	-1	-1	2	2	1	0	-1	-1
0.05-0.005.....	19	18	16	-1	-2	-3	19	18	17	-1	-1	-2
0.005-0.001.....	16	10	8	-6	-2	-8	18	13	9	-5	-4	-9
Less than 0.001.....	43	50	55	+7	+5	+12	41	47	53	+6	+6	+12

<sup>1</sup> Amount found is given as percentage of total weight of soil; differences are in percentage points.

it is believed that little of this effect is reflected in these data.

Since dispersant concentration is the only variable involved in each set of figures, decreases in the coarser sizes are attributed to the breakdown of true aggregates. When the dispersant concentration is increased, the disaggregation increases until a maximum dispersion is reached at the optimum concentration. Differences in the degree of dispersion of the same soil with different dispersants are accordingly attributable to the greater ability of the better dispersant to disintegrate the aggregate structures and to maintain a condition of deflocculation in the resulting suspension.

The data in table 8 show the effects of the two mechanical dispersion devices on the percentages of the several particle-size groups in two soils, Cecil and Sharkey. The values given are for dispersion treatments of 15 minutes with the air-jet dispersion cup and 1-minute with the motor-driven stirrer, using, for both treatments, 20 ml. of 0.2N sodium metasilicate solution, in accordance with the standard AASHTO method T 88-49. This amount of the silicate solution also corresponds to the optimum concentration for both soils. The degree of dispersion produced by the air-jet dispersion cup treatment is much greater than that by the motor-driven stirrer, as indicated by the values of the less-than-0.001-mm. group. In the Cecil soil, the gain of this group is derived from small reductions of all

of the coarser groups, whereas most of the reduction in the Sharkey soil occurs in the 0.05-0.005-mm. group.

Although an extensive study of the mineralogical nature and the physical and chemical properties of the difficultly dispersed soil constituents was beyond the scope of the present investigation, studies of this sort offer interesting possibilities. It would be of value to know the identity and quantity of the clay or other active fine material which is "bound" in difficultly dispersed aggregates of silt and sand sizes; whether physical properties such as plasticity and volume change of the aggregated clay material are inhibited, and if so, to what extent the properties of the whole soil are affected; and the basic nature of the

binding forces in inorganic soils and why and to what extent they are effective in soils of different origin.

### Sediment Volumes and pH Values

During the sedimentation of the soil suspensions, a large number of measurements of sediment volumes and pH values (indicating degree of acidity) and observations of colloidal activities were made. Since the measurements do not constitute a thorough and specific study of any of these effects, the bulk of the data is therefore not included in this report. However, some of the findings warrant a brief mention.

Sediment volumes measured in suspensions (after a 24-hour sedimentation period) pre-

**Table 8.—Comparison of percentages of various size-groups from dispersions with the air-jet dispersion cup and the motor stirrer<sup>1</sup>**

Size-group (mm.)	Amount found (percent of total weight of soil)					
	Cecil soil (No. 2)			Sharkey soil (No. 5)		
	Air-jet dispersion cup	Motor stirrer	Difference <sup>2</sup>	Air-jet dispersion cup	Motor stirrer	Difference <sup>2</sup>
2-0.42.....	8	9	-1	6	6	0
0.42-0.25.....	4	6	-2	3	3	0
0.25-0.074.....	17	20	-3	14	16	-2
0.074-0.05.....	4	5	-1	3	6	-3
0.05-0.005.....	23	24	-1	26	35	-9
0.005-0.001.....	11	14	-3	20	21	-1
Less than 0.001.....	33	22	+11	28	13	+15

<sup>1</sup> Using the optimum concentration of sodium metasilicate (20 ml. of 0.2N solution).

<sup>2</sup> Difference in percentage points.

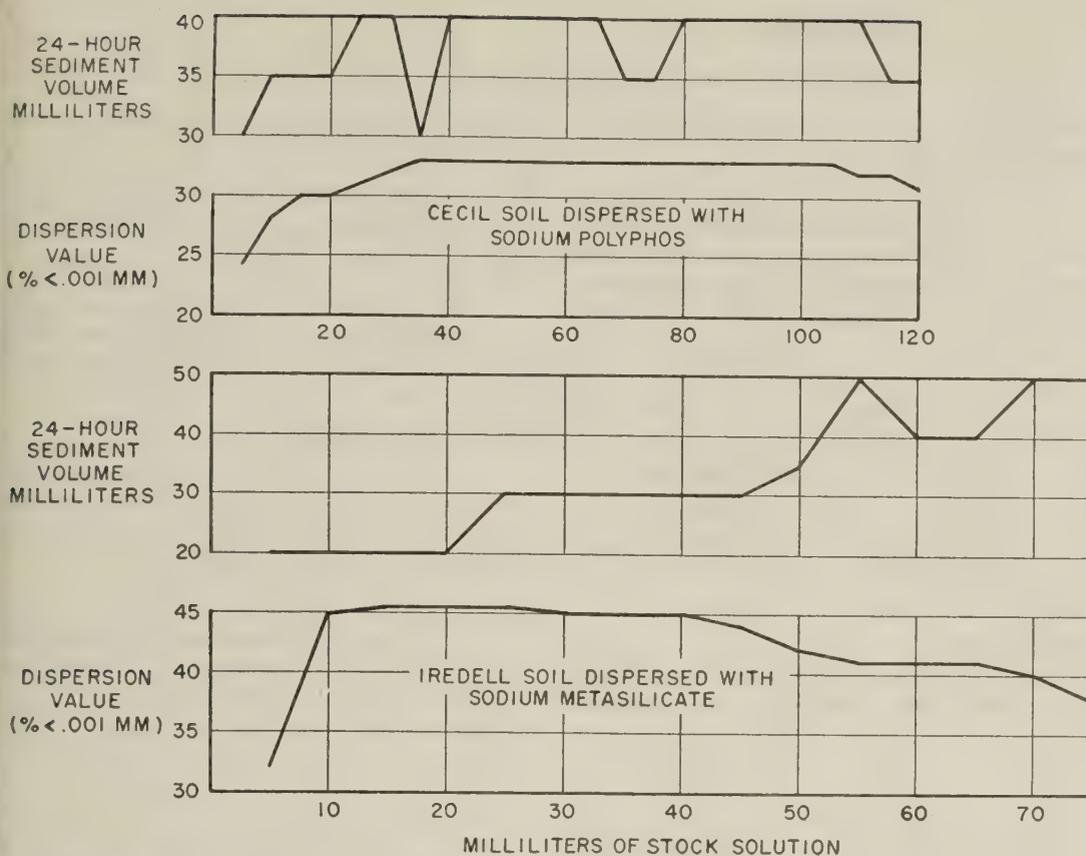


Figure 2.—Effect of dispersion value on sediment volume with Cecil and Iredell soils.

pared with various concentrations of a dispersing agent are not a good index of the degree of dispersion. In the two examples given in figure 2, contrary to what might be expected, the sediment volume is small at a low degree of dispersion, and increases as dispersion improves. Although the relation varied with different soils and dispersing agents, very similar results were obtained in many of the other tests.

Colloidal effects such as flocculation and gelation, which interfere with the analytical results, are often unnoticed unless they are strongly manifested. However, less evident ones can often be detected by simple tests. The absence of a sharp line of demarcation between the sediment and the suspension, after a brief settling period, invariably indicates an abnormality of the deflocculation process. This observation can be made long before any pronounced flocculation or gelation occurs. A gelled volume in the lower part of the suspension is sometimes observed as a dark layer, but is often obscured by the color of the soil. It can more readily be detected by the pearly iridescence which results when the suspension is caused to rotate in the cylinder by means of a glass rod. With normal deflocculation, stirring produces a pearly effect of light and dark streaks throughout the suspension from the reflection of light by the flat sides of platy particles which are highly oriented by the rotation. If gelation is present in any part of the suspension, this phenomenon is reduced or obscured in the affected area. Gelation is also indicated when, after a reading has been made and the hydrometer removed from the suspension, the liquid remaining on the hydrometer flows off

as a film; or when, upon pouring the suspension into a No. 200 or No. 270 sieve for separation and washing of the coarse soil fractions, a restriction of the flow of the suspension through the sieve is noted. Although any of these qualitative tests may be used to advantage, the analytical procedure should include as a minimum requirement a routine examination of each suspension under bright illumination so that the appearance of the demarcation line and any pearly iridescent effect can be observed.

Some of the data from pH measurements of suspensions obtained by the dispersion of each of soils 1-6 with each of the phosphates are given in table 9. The two pH values shown for each soil-dispersant combination are those which occurred at the high and low extremes of the practical concentration range of the dispersing agent when used with that soil.

The figure in parentheses adjacent to each pH range is the deviation (in percentage points) of the dispersion value obtained in that range from the maximum obtained for the soil.

As in the previous discussions, the data for soils 1-5 are considered separately from those for soil 6, the laterite. With the group of soils 1-5, the pH range in which each dispersing agent is most effective, is different for different soils. For example, the pH ranges for dispersant B are within narrow limits (6.6 to 7.5) and are nearly identical; those for dispersant E consist of somewhat higher values and are broader in scope (6.8 to 9.1). It is further observed that for any one of the soils of this group, both a high degree of dispersion and good stability of the suspension can occur at several different pH ranges, depending on the dispersant used. However, suspension failure (flocculation of soils 2, 3, and 4 with dispersant C) can also occur over very broad pH ranges which include values at which suspensions are successfully maintained by other dispersants. The stock solutions of the better dispersants, B, E, F, and G, for soils 1-5 have pH values between 6.7 and 8.8; that of D, which is less effective, is 10.0; and those of C and H, which are least effective, are 11.3 and 4.3.

In contrast to soils 1-5, the laterite (6) is effectively dispersed only under conditions of high pH; its deviations are lowest (best dispersion) at pH values of 9.2 and above with dispersants C and D, intermediate in the range 8.1-9.0 with dispersant E, and highest (poorest dispersion) in the range 6.2-6.7 with dispersant H.

Puri has presented data (9) showing that many soils are most highly dispersed at a pH of 11.2 when sodium hydroxide is used as the dispersant and the soil samples are pretreated to convert them to the H-form. The data in table 9 are at variance with Puri's findings, since they were obtained using soil samples in their natural cationic state, and phosphate materials which are more effective at lower pH values. The present investigation was concerned with dispersion techniques which provide a high degree of dispersion without time-consuming pretreatments of the soil samples.

Table 9.—pH values in suspensions of soils 1-6 with the phosphates

Dispersing agent		pH ranges <sup>1</sup> and deviations <sup>2</sup> from the maximum dispersion values for soil —					
Identification	pH of stock solution	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
B	6.8	6.6-7.0 (2)	7.3-7.3 (0)	7.4-7.4 (0)	7.1-7.2 (1)	7.2-7.5 (2)	7.4-7.4 (0)
C	11.3	8.7-9.2 (5)	6.7-10.7 F	7.6-11.1 F	7.2-11.4 F	9.4-10.9 (6)	9.5-11.3 (0)
D	10.0	8.2-8.7 (4)	6.2-9.0 (2)	8.6-9.5 (2)	8.5-9.3 (3)	8.8-9.3 (3)	9.2-9.4 (1)
E	8.8	7.2-7.6 (3)	6.8-8.4 (1)	7.3-9.1 (1)	7.2-8.9 (1)	7.9-8.8 (1)	8.1-9.0 (5)
F	7.6	6.7-7.2 (2)	7.8-8.1 (0)	8.1-8.2 (0)	7.3-8.1 (0)	7.4-7.8 (3)	7.9-8.1 (0)
G	6.7	6.6-6.8 (1)	5.8-6.8 (3)	7.2-7.3 (2)	6.6-7.3 (2)	7.0-7.3 (2)	7.3-7.4 (13)
H	4.3	5.6-6.1 (3)	6.0-6.2 (2)	ND	ND	ND	6.2-6.7 (19)
pH of soil (1-1 mix)		5.7	4.6	5.1	4.9	6.7	4.9

<sup>1</sup> The two values shown for each soil-dispersant combination are those which occurred at the high and low extremes of the practical concentration range of the dispersing agent when used with that soil.

<sup>2</sup> All values in parentheses are deviations (in percentage points) of the given dispersion value from the maximum value obtained for that soil.

F=flocculation at all concentrations and over the pH range cited; ND=pH not determined.

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		PROGRAMMED ONLY			PLANS APPROVED, CONSTRUCTION NOT STARTED			CONSTRUCTION UNDER WAY			TOTAL		
		Total Cost	Federal Funds	Miles	Total Cost	Federal Funds	Miles	Total Cost	Federal Funds	Miles	Total Cost	Federal Funds	Miles
Alabama	\$6,079	\$9,554	\$4,923	310.0	\$12,137	\$6,268	216.9	\$43,978	\$21,900	477.2	\$65,669	\$33,091	1,004.1
Arizona	2,027	5,270	3,681	125.5	1,421	1,058	33.5	6,468	4,518	94.6	13,159	9,257	253.6
Arkansas	3,784	10,172	5,847	409.8	5,754	2,831	171.4	18,220	9,079	411.3	34,146	17,757	992.5
California	1,432	19,038	8,626	159.3	26,413	11,980	77.8	97,492	48,043	271.0	142,943	68,649	508.1
Colorado	9,201	4,033	2,258	39.2	333	187	1.6	18,629	10,183	206.7	22,995	12,628	247.5
Connecticut	8,968	2,652	1,326	7.9	4,600	2,122	6.1	5,090	2,554	12.6	12,342	6,002	26.6
Delaware	1,566	2,530	1,290	9.8	2,994	1,841	8.1	5,551	2,763	34.3	11,075	5,894	52.2
Florida	4,163	24,173	12,247	389.4	5,428	2,742	62.2	20,863	11,016	332.4	50,464	26,005	784.0
Georgia	13,358	10,134	5,211	196.0	7,073	3,497	141.5	37,823	17,740	545.4	55,030	26,448	882.9
Idaho	1,877	4,662	3,012	99.4	6,629	4,135	97.4	15,796	9,798	287.5	27,087	16,945	484.3
Illinois	12,770	34,465	18,472	345.7	10,279	5,194	121.7	77,294	40,731	485.7	122,038	64,397	953.1
Indiana	7,204	44,193	23,104	184.9	19,956	10,078	97.8	22,168	11,509	143.6	86,317	44,691	426.3
Iowa	4,248	11,172	6,151	395.4	8,964	4,807	380.2	26,954	14,534	849.3	47,090	25,492	1,624.9
Kansas	6,384	10,312	5,200	804.1	6,622	3,325	495.8	19,197	9,677	720.0	36,131	18,202	2,019.9
Kentucky	5,803	9,995	5,312	102.6	7,498	3,752	94.0	23,885	12,406	272.3	41,378	21,470	468.9
Louisiana	3,856	15,302	7,640	115.2	15,901	6,890	92.3	24,326	11,755	99.1	55,529	26,265	306.6
Maine	3,960	5,455	2,804	45.4	509	255	5.8	12,985	6,424	97.5	18,949	9,483	148.7
Maryland	6,466	12,976	6,739	81.5	5,027	2,790	61.5	9,242	4,663	43.8	27,245	14,192	186.8
Massachusetts	2,337	12,285	6,133	22.1	1,685	988	.3	57,080	26,825	42.8	71,050	33,946	65.2
Michigan	3,809	39,952	19,995	496.4	16,060	8,034	355.8	40,385	19,199	271.3	96,397	47,228	1,123.5
Minnesota	4,573	10,374	5,373	758.7	12,832	6,668	928.2	25,659	13,349	614.4	48,865	25,390	2,301.3
Mississippi	4,568	11,278	5,721	353.8	6,229	3,198	224.8	22,433	11,290	601.7	39,940	20,209	1,180.3
Missouri	10,060	17,670	9,111	861.4	8,697	4,621	319.4	57,147	20,293	581.1	83,514	42,025	1,761.9
Montana	5,137	15,804	9,553	349.9	5,241	3,238	191.9	22,258	13,645	452.8	43,303	26,436	994.6
Nebaska	9,003	22,797	11,892	895.9	5,219	2,823	83.4	17,468	9,339	511.1	45,484	24,054	1,490.4
Nevada	6,260	2,548	2,185	81.6	782	653	44.0	8,732	7,249	236.0	12,062	10,087	361.6
New Hampshire	2,192	4,376	2,188	26.8	1,636	812	6.3	7,150	3,635	38.9	13,162	6,635	72.0
New Jersey	10,191	4,233	2,117	51.7	1,976	949	5.5	23,023	8,856	15.8	29,232	11,922	73.0
New Mexico	2,452	5,264	3,329	129.0	2,484	1,575	42.4	9,848	6,072	227.1	17,596	10,976	398.5
New York	5,843	80,463	41,956	117.6	31,834	16,370	77.4	189,531	90,234	488.7	301,828	148,560	683.7
North Carolina	5,252	23,363	11,651	441.2	6,560	3,117	140.1	38,723	18,055	466.8	68,646	32,823	1,048.1
North Dakota	3,569	4,677	2,352	769.7	6,440	3,220	497.0	9,976	4,993	708.8	21,093	10,565	1,975.5
Ohio	7,308	25,751	13,000	102.9	19,313	8,899	40.0	85,755	39,863	162.2	130,819	61,762	305.1
Oklahoma	7,899	21,116	11,405	321.2	6,234	3,275	149.8	16,523	8,771	234.8	43,873	23,451	705.8
Oregon	2,622	3,409	1,983	23.3	2,019	1,213	73.0	15,726	9,520	209.1	21,154	12,716	305.4
Pennsylvania	3,898	23,255	11,602	16.9	36,127	17,888	56.9	95,817	46,820	214.4	155,199	76,310	288.2
Rhode Island	1,094	8,118	4,059	27.4	994	496	8.9	7,854	3,925	36.5	16,966	8,480	72.8
South Carolina	6,265	8,792	4,701	178.7	3,801	1,993	141.9	16,081	8,171	525.5	28,674	14,865	846.1
South Dakota	1,978	12,627	7,163	653.6	1,853	1,157	117.7	8,869	5,048	480.9	23,349	13,368	1,252.2
Tennessee	7,073	14,438	7,172	372.8	7,431	3,724	201.3	36,654	16,733	494.3	58,523	27,629	1,068.4
Texas	13,658	9,691	5,054	88.0	15,991	7,905	445.9	66,733	35,614	1,121.3	92,415	48,573	1,655.2
Utah	506	5,260	4,057	78.7	3,358	2,509	97.3	9,117	6,978	99.1	17,735	13,544	275.1
Vermont	1,268	2,902	1,459	33.7	1,130	554	7.7	8,945	4,515	65.7	12,977	6,528	107.1
Virginia	4,387	14,635	7,108	249.7	6,318	3,142	129.7	29,318	13,915	183.0	50,271	24,165	562.4
Washington	1,358	12,911	6,767	120.6	7,248	3,627	169.3	19,375	10,275	189.5	39,534	20,669	479.4
West Virginia	6,144	10,006	5,055	56.5	1,976	1,000	3.1	16,243	8,094	47.5	28,225	14,149	107.1
Wisconsin	3,685	17,987	9,471	176.3	8,810	4,386	193.1	27,900	13,809	361.4	54,697	27,666	730.8
Wyoming	544	5,638	3,669	108.5	1,029	667	20.0	9,654	6,050	250.3	16,321	10,426	378.8
Hawaii	3,184	932	466	2.0	634	309	2.2	11,969	5,651	15.5	13,535	6,426	19.7
District of Columbia	4,298	6,793	3,157	2.9	996	498	2.3	10,569	5,081	1.4	18,358	8,736	6.6
Puerto Rico	5,607	6,588	3,124	38.5	5,239	2,182	9.2	13,937	6,589	40.9	25,764	11,895	88.6
TOTAL	261,168	702,021	367,871	11,829.1	385,714	195,442	6,951.4	1,522,413	765,789	15,374.9	2,610,148	1,329,102	34,155.4

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